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Original Article

Effect of laser engraving on shear bond strength of polyetheretherketone to indirect composite and denture-base resins

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Thermocycling

Abstract *Background/purpose:* Polyetheretherketone (PEEK) is a highly sought-after thermoplastic due to its exceptional mechanical properties and biocompatibility. However, bonding PEEK to indirect composite resin (ICR) or denture-based resin (DBR) can be challenging. Laser engraving technology has shown potential to improve bonding for other materials; thus, this study aims to evaluate its effectiveness for PEEK.
Materials and methods: The experiment involved preparing ingot-shaped PEEK samples, which were then categorized into four groups based on the treatment method employed: without treatment, air abrasion, sulfuric acid etching, and laser engraving (LS). Subsequently, the samples were bonded to ICR or DBR, and their shear bond strength (SBS) was tested with or without thermocycling using a universal testing machine. Furthermore, the failure mode was observed, with statistical analyses conducted to compare the results.
Results: The grid-like microslit structure of LS group displayed the highest SBS for bonding PEEK to ICR or DBR ($P < 0.05$). During the bonding of PEEK to ICR, resin residue and penetration into the microslits were frequently observed in the LS group, indicating cohesive failure. However, when PEEK was bonded to DBR, mixture failure was frequently observed without

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thermocycling. After thermocycling, only the LS group showed cohesive failure, while the majority of specimens exhibited mixture failure.
Conclusion: Laser engraving significantly improves the SBS between PEEK and both ICR and DBR. Furthermore, it was observed that resin had penetrated the microslits, indicating that laser engraving has great potential as a surface treatment method.

Introduction

Polyetheretherketone (PEEK) is a high-performance semi-crystalline thermoplastic polymer with a linear structure comprising aromatic rings bonded with ether (R–O–R′) and ketone (R–C(=O)–R′) functional groups. PEEK possesses various qualities, including high mechanical strength, chemical stability, ease of processing, dimensional stability at high temperatures, and ecological compatibility.[1–3] As a result, it is a highly sought-after material in various industries, including the automotive sector, where it is used for manufacturing engine parts and electronic components. Additionally, PEEK has garnered increasing interest in the biomedical field owing to its excellent biocompatibility, human bone-like elasticity, low plaque affinity, and minimal allergy-causing potential.[4–6] Furthermore, due to its other desirable properties, such as radiation transparency, PEEK is emerging as a promising substitute for metallic biomedical materials, particularly in dental applications.[7,8] Prior research has demonstrated that PEEK has been utilized in dental restorations, including crowns, implant abutments, and removable denture frameworks (such as clasps).[9–11]

Although compatible with computer-aided design and manufacture (CAD/CAM) processing, PEEK requires additional veneering since its native gray color is opaque and lacks aesthetic appeal. When used as a framework for removable dentures, it is necessary to bond PEEK to denture-based resin (DBR). For areas needing aesthetics, such as the anterior teeth, it is necessary to veneer PEEK with indirect composite resin (ICR).[5,12] However, reports indicate that establishing a strong bond between PEEK and DBR can be challenging due to the high water absorption and high thermal expansion coefficient of DBRs. Moreover, it was also highlighted that bonding between PEEK and ICR is crucial for clinical application.[12–14]

The bonding performance of PEEK can be improved through the combination of chemical and mechanical surface treatments.[15,16] Chemical treatment involves using primers to create chemical adhesion, while mechanical treatment roughens the surface and increases the surface area.[1,3–5,12,17] Research has indicated that adhesive primers containing methyl methacrylate (MMA) are effective for PEEK bonding.[3,5,17] For mechanical treatments, several studies have reported that air abrasion improves bond strength.[18–20] Although etching with concentrated sulfuric acid (H_2SO_4) exhibits high bond strength, it is considered unsafe for clinical practice, and other effective methods are needed.[18–22] While some investigations have reported high bond strength for etching PEEK with piranha solution or plasma treatment, certain studies have found that it does not significantly impact bonding with resin cement.[4,12,18,23] Moreover, recent research has revealed that UV irradiation below the ablation threshold can increase the bond strength of PEEK by up to tenfold.[24,25]

In 2012, the use of laser engraving technology to create micro-mechanical retention was first proposed by the Dr. Shimoe's group to improve the shear bond strength (SBS) of porcelain fused to zirconia (Zr).[26] Subsequently, multiple studies have confirmed that laser engraving can significantly increase the SBS of Zr to DBRs or ICRs by creating micro-slits prior to bonding, which increases the surface area.[27–31] Accordingly, it is believed that laser engraving could improve the surface characteristics of PEEK, enhance mechanical bonding, and ultimately increase the SBS.[26–31] This study aims to evaluate the impact of grid-like microslits created by laser engraving on PEEK's surface and compare it with other mechanical surface treatments for bonding PEEK to DBRs or ICRs. The null hypothesis of this study is that laser-engraved grid-like microslits do not significantly improve the shear bond strength of PEEK compared to other surface treatments.

Materials and methods

Specimen fabrication and surface treatment

Table 1 lists the materials used in this study, with Fig. 1 showing the experimental process. Ingot-shaped PEEK samples (Polyplastics-Evonik Corporation Ltd., Osaka, Japan) with a diameter of 10 mm and a thickness of 3.0 mm were designed and prepared (n = 160). All the samples were ground flat using 600-grit abrasive papers (Sankyo-Rikagaku Co., Ltd., Saitama, Japan). Next, the samples were randomly divided into four groups and then subjected to surface treatment using the following methods (n = 40 per group): without any treatment, denoted as NT; air abraded with 50 μm alumina particles (Renfert GmbH, Hilzingen, Germany) under a blasting pressure of 0.25 MPa for 10s using a grit blaster (Renfert GmbH, Hilzingen, Germany), denoted as AB; treated with 100 mL of 95% H_2SO_4 on the sample surfaces for 60s, followed by careful rinsing with deionized water for 60s and eventually cleaned using an ultrasonic cleaner for 10 min, denoted as SA; and laser-engraved grid-like micro-slits with a width, pitch, and depth of 50 μm on the sample surfaces, denoted as LS. After surface treatment, the samples were cleaned using a dental steamer and then allowed to air-dry.

Table 1 Materials used and their specifications.

Identification		Main composition[a]	Manufacturer	Lot No.
VESTAKEEP® DC4450R		poly (ether-ether-ketone)	Polyplastics-Evonik Corporation Ltd., Osaka, Japan	57781699
cobra		Al_2O_3, SiO_2	Renfert GmbH, Hilzingen, Germany	700388
sulfuric acid		95% H_2SO_4	Katayama Chemical Industries Co., Ltd., Hyogo, Japan	N0555
Adhesive primer				
visio.link		MMA, PETA	Bredent medical GmbH & Co. KG, Senden, Germany	193211
Indirect composite resin (ICR)				
Gradia	FO	GC Corp., Tokyo, Japan	GC Corp., Tokyo, Japan	1811131
	OA3	UDMA, silica nano powder		1805181
	DA3	UDMA, inorganic-organic composite filler, silica nano powder, glass powder		1901211
Denture based resin (DBR)				
PalaXpress® Ultra		Powder: PMMA Liquid: MMA	Kulzer Japan Co., Ltd., Tokyo, Japan	012030 010233

[a] The main composition is based on the information provided by the manufacturer; MMA, methyl methacrylate; PETA, pentaerythritol triacrylate; PMMA, poly (methyl methacrylate); UDMA: urethane dimethacrylate.

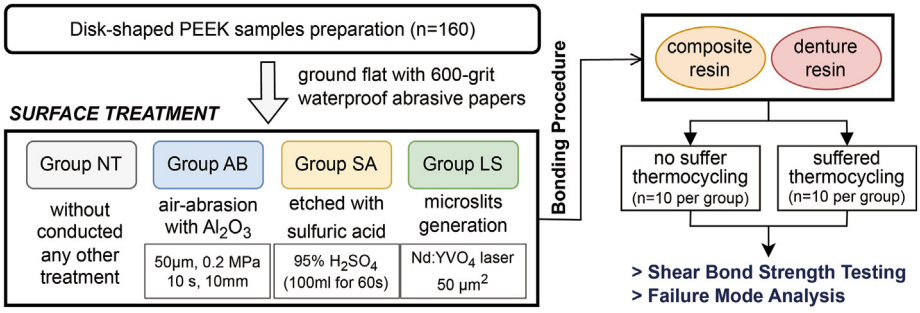

Figure 1 Experimental diagram.

Bonding procedure of polyetheretherketone with resins

To define the bonding area, a double-sided adhesive tape with a circular hole measuring 5 mm in diameter was affixed onto the PEEK sample. Next, a visio.link primer (Bredent medical GmbH & Co. KG, Senden, Germany) was applied to the bonding area, followed by light-curing for 90 s. The PEEK samples were bonded with the ICR (GC Corp., Tokyo, Japan) using the following procedure: A thin layer of opaque resin and an additional opaque material were applied to the primed surface and underwent 60 s of light-polymerization. Once the curing process was complete, a brass mold with an internal diameter of 6 mm, a length of 2 mm, and a height of 1 mm was fixed in place, filled with dentin shade of the ICR (GC Corp.), and light-polymerized for 180 s. To bond PEEK samples with the DBR (Kulzer Japan Co., Ltd., Tokyo, Japan), a brass mold with the same dimensions as mentioned above was used to define the bonding area. Next, the liquid and powder of the DBRs (Kulzer Japan Co., Ltd.) were mixed according to the manufacturer's recommended ratio and poured into the brass mold. Eventually, the samples were heat-polymerized at 55 °C and 0.2 MPa for 30 min using a pressure vessel (Kulzer Japan Co., Ltd., Tokyo, Japan). After the completion of the bonding process, all the samples (n = 20) were immersed in distilled water at 37 °C for 24 h, and half of these specimens (n = 10) were placed in a thermocycling apparatus (Nissin Seiki Co. Ltd., Hiroshima, Japan) and cycled between 4 °C and 60 °C in water with a 1 min dwell time per bath for 20,000 cycles.

Shear bond test and failure analysis

The samples bonded with ICRs or DBRs were placed in a testing jig, and a universal testing machine (Shimadzu Corp., Kyoto, Japan) was used to perform the shear bond test. Shear force was applied to the adhesive interface until fracture occurred at a crosshead speed of 0.5 mm/min. The SBS was calculated after the test by dividing the peak load (in N) by the bonding area (in mm^2). To determine the mode of failure, the fractured interfaces were observed with an optical microscope (Inoue Attachment Corp., Kanagawa, Japan) at an original magnification

of × 8. The mode of failure was classified as an adhesive failure at the PEEK-resin material interface, mixture failure of cohesive and adhesive, and cohesive failure within the resins. Representative specimens were observed by scanning electron microscopy (SEM) (Keyence Corp., Osaka, Japan).

Statistical analysis

The Shapiro–Wilk and Levene's tests were conducted to confirm the normality and homogeneity of all the obtained values. As all data in this experiment were normally distributed, statistical comparisons of the SBS results for various groups were performed using the two-way analysis of variance (ANOVA) method and Scheffé's test for multiple comparisons. The statistical significance level was set at 0.05. All analyses were performed using SPSS software (IBM SPSS version 19.0, Armonk, NY, USA).

Results

Surface characteristics of different surface treatment

Fig. 2 displays the SEM image of the PEEK surface after preprocessing. The NT group shows a uniform line grinding pattern, while the AB group presents an irregular surface morphology resulting from air-abrasion. The SA group exhibits a relatively smooth surface with some visible pores. In contrast, the LS group displays a distinctive surface morphology, which is characterized by regular grid-like microslits created through laser engraving.

Polyetheretherketone bonded to indirect composite resin

Table 2 summarizes the results of SBS testing under various conditions. The LS group demonstrated the statistically highest SBS ($P < 0.05$), while the NT group exhibited the statistically lowest SBS ($P < 0.05$). However, no significant difference was observed between the AB and SA groups ($P > 0.77$). Comparison of pre- and post-thermocycling results revealed no significant reduction in SBS in any of the groups ($P > 0.29$), except for the LS group, which showed a significant increase after thermocycling ($P < 0.05$). Cohesive failure was observed in the AB, SA, and LS groups, whereas the NT group exhibited adhesive failure in all cases (Table 2). The SEM images presented in Fig. 3 showed the presence of resin residue on the surfaces of the AB and LS groups, regardless of thermocycling. Furthermore, the resin was found to penetrate the grid-like microslits of the LS group, indicating cohesive failure. In contrast, no resin was detected on the surfaces of the NT group, indicating adhesive failure.

Polyetheretherketone bonded to denture-based resin

As shown in Table 2, both the AB and LS groups demonstrated significantly higher SBS than the NT group, regardless of whether they underwent thermocycling ($P < 0.05$).

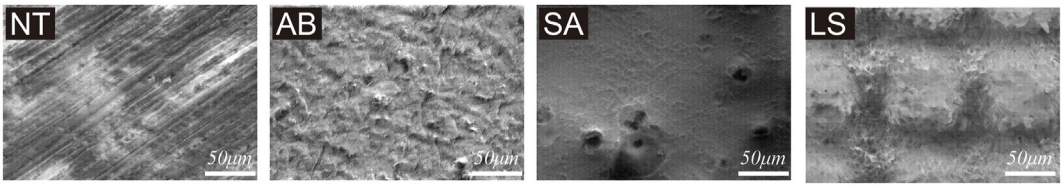

Figure 2 Surface microstructure morphology of PEEK samples after surface treatment (×500).

Table 2 The bonding strength (MPa) and failure modes results.

Groups	Surface treatment	0 thermal cycle				20,000 thermal cycles				S
		SBS	A	M	C	SBS	A	M	C	
ICR	NT	10.05 ± 3.45^{a}	10	0	0	12.63 ± 5.99^{A}	10	0	0	
	AB	22.79 ± 4.27^{b}	0	0	10	21.65 ± 2.78^{B}	0	0	10	
	SA	21.04 ± 2.49^{b}	0	0	10	21.05 ± 3.66^{B}	0	0	10	
	LS	30.76 ± 1.54^{c}	0	0	10	34.75 ± 2.45^{C}	0	0	10	S
DBR	NT	9.79 ± 1.88^{a}	3	7	0	4.03 ± 1.36^{A}	0	8	2	S
	AB	17.76 ± 5.24^{b}	6	4	0	7.79 ± 0.93^{B}	0	9	1	S
	SA	$15.76 \pm 5.49^{a,b}$	0	10	0	$5.76 \pm 3.19^{A,B}$	0	9	1	S
	LS	35.34 ± 3.05^{C}	0	7	3	25.06 ± 3.48^{C}	0	10	0	S

Groups, indirect composite resin (ICR) and denture-based resin (DBR). Surface treatment methods, without any treatment (NT), air abraded with alumina particles (AB), treated with 95% H_2SO_4 (SA), and laser-engraved (LS); Shear bond strength (SBS) represented as mean ± standard deviation. Within the same column, different letters indicate groups that are statistically different ($P < 0.05$). Failure mode dived as adhesive failure (A), mixture of cohesive and adhesive failure (M), and cohesive failure (C). S: significant difference between 0 and 20,000 thermal cycles ($P < 0.05$). Reduction, rate of SBS reduction.

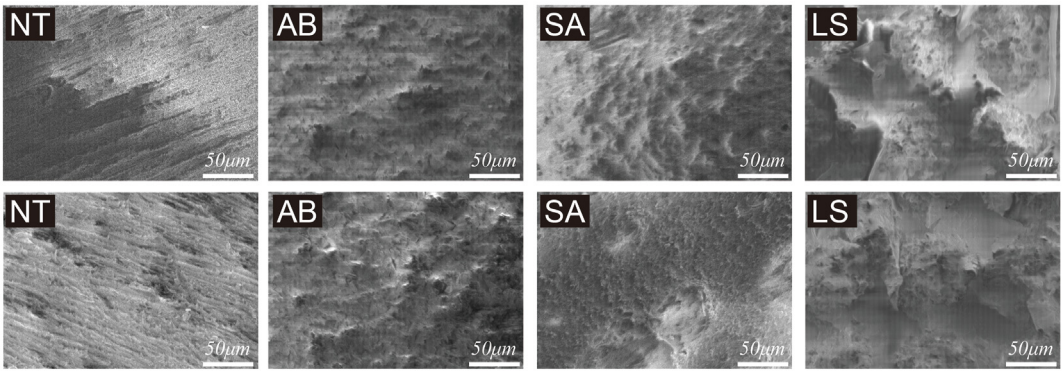

Figure 3 Debonded fracture interface of composite resins and PEEK. The upper row is the group that did not encounter thermocycling groups (0 cycles), and the lower row is the group that encountered thermocycling groups (20,000 cycles).

Specifically, the LS group exhibited considerably higher SBS even after thermocycling ($P < 0.05$). Furthermore, when comparing pre- and post-thermocycling results, a significant reduction in SBS was observed in all groups that underwent thermocycling ($P < 0.05$). In specimens without thermocycling, mixture failure was commonly observed, with cohesive failure only observed in the LS group. Additionally, adhesive failure was observed in the NT and AB groups without thermocycling. However, after undergoing thermocycling, most of the specimens exhibited mixture failure (Table 2 and Fig. 4).

Discussion

Establishing an effective bond between PEEK and DBR or ICR is a critical factor in evaluating the clinical performance of dental applications. Sufficient shear bond strength (SBS) is essential for ensuring long-term survival without failure. However, the chemical stability of the bond presents a challenge. To overcome this issue, approaches such as mechanical retention and the use of adhesive primers are employed.[16] In this study, an adhesive primer containing MMA was applied (Table 1),[3,5] and the effect of different mechanical surface treatments on the bond strength of PEEK was evaluated.[5,32] The experiment results showed that the laser-engraved grid-like microslits demonstrated superior bonding performance, leading to the rejection of the null hypothesis.

The LS group demonstrated significantly higher SBS values ($P < 0.05$) than the AB group for bonding with ICR, regardless of thermocycling. However, no significant difference was observed for the SA group ($P = 0.996$). SEM images (Fig. 2) show that higher SBS corresponds to more surface irregularities, grooves, and cracks caused by mechanical treatment. The LS group had the highest SBS due to deeper irregularities from the laser-engraved grid-like microslits, resulting in strong mechanical interlocking. Previous studies have shown that pretreatment with H_2SO_4 or piranha solution yields better SBS than air abrasion.[4,13] However, the adhesive primers used in these studies did not contain MMA and were not light-activated. This suggests that the light-cured adhesive primer may have covered the porous surface created by the H_2SO_4 treatment, leading to lower bond strength in the SA group than in the AB group. When the groups were compared under thermocycling, only the LS group showed a significantly higher SBS ($P < 0.05$). A previous study demonstrated that using an adhesive primer when bonding PEEK to resin did not have a negative impact on the SBS results following thermocycling.[33] The durable bond strength of PEEK against mechanical stress caused by thermocycling is attributed to the presence of PETA in the adhesive primer and the absence of acid groups.[9] The laser-engraved grid-like microslits on the PEEK surface provided a

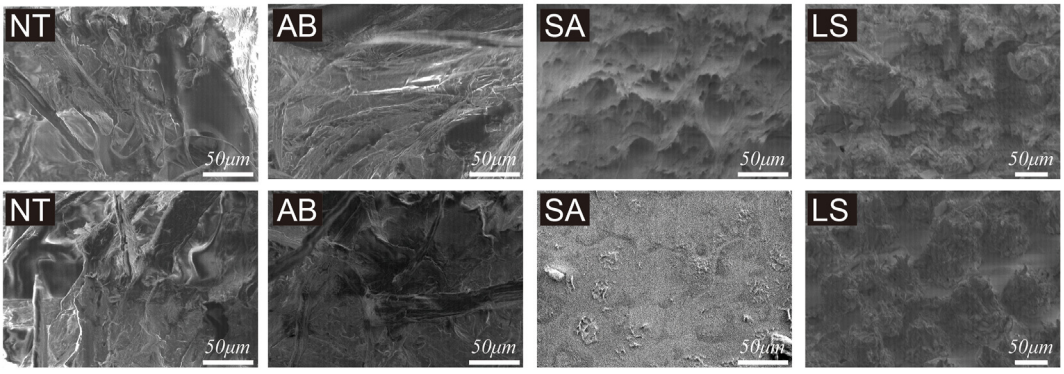

Figure 4 Debonded fracture interface of denture-based resins and PEEK. The upper row is the group that did not encounter thermocycling groups (0 cycles), and the lower row is the group that encountered thermocycling groups (20,000 cycles).

strong defense against delamination caused by thermal expansion of the PEEK and ICR, as only the LS group showed a considerably higher value after thermocycling ($P < 0.05$). When observing the debonded surfaces after the shear test, all groups except for the NT group showed cohesive failure, and SEM images revealed residual resin on the debonded surfaces of PEEK (Fig. 3). The literature suggests that the use of an adhesive primer containing MMA on a surface with fine retention, such as achieved through blasting or plasma treatment, can be effective for bonding PEEK and resin. It is speculated that using the adhesive primer contributes to a stronger bond.[18,23]

The AB group exhibited a significantly higher bond strength than the NT group when bonding PEEK with DBR, regardless of whether thermocycling was performed ($P < 0.05$). As previously reported, air abrasion increases the surface area of PEEK, enabling effective mechanical retention.[13,20] Literature indicates that the surface treatment with H_2SO_4 significantly corrodes the surface of PEEK, creating roughness and increasing its porosity.[19,21] This leads to an increase in the surface area of PEEK, similar to air abrasion, and is suspected to facilitate effective mechanical retention. While other studies have reported that surface treatment with H_2SO_4 demonstrates bond strength comparable to or higher than that of air abrasion,[20] no significant difference in bond strength was observed between the SA and NT groups in this study. This may be due to the fact that the DBR used in this study had poor flowability, which did not allow it to penetrate the porous roughness created by pretreatment with sulfuric acid. Therefore, it is suspect that pretreatment with H_2SO_4 did not function effectively in terms of mechanical retention compared to air abrasion. However, it is possible to improve the bond strength using effective combinations of PEEK types or primers. This is because significant variations in bond strength have been reported even under the same surface treatment conditions.[4,5] The LS group exhibited significantly higher SBS ($P < 0.05$) than the AB and SA groups, regardless of whether thermocycling was performed. This could be attributed to the grid-like microslits created by laser engraving, which increased the contact area of the PEEK surface and enhanced mechanical retention, ultimately resulting in improved bond strength. Furthermore, compared to the surface roughness generated by air abrasion or H_2SO_4 treatments, the grid-like microslits engraved by laser have larger irregularities and increase the surface area of PEEK, which may enhance mechanical retention. Previous studies have reported that pretreatment with acid can increase the number of functional groups on the surface of PEEK. This is due to the acid bonding with the carbonyl and ether groups of PEEK, providing functional groups on the surface of PEEK and thereby improving the bond strength.[24] Acid etching can chemically modify the surface of PEEK, enhancing its reactivity with resin-based adhesive primers and increasing the functionality of the PEEK surface.[12,24] When used in combination with acid etching, an adhesive primer containing multi-functional methacrylate can establish a strong chemical bond with PEEK.[12] Regarding the debonded surface, most of the samples displayed mixed fractures. However, adhesive failures were observed in the NT and AB groups without thermocycling. It is believed that the adhesive failures occurred due to differences in water absorption and thermal expansion coefficient of the resin, causing delamination at the surface of PEEK.

The current study proposes that laser-engraved grid-like microslits effectively improve the bonding performance between PEEK and ICR or DBR compared to other treatments. However, to apply laser engraving in clinical settings, various issues, such as the cost and processing time, along with the effects on the bond strength of PEEK, still need to be considered. Additionally, since this study did not examine the shear direction relative to the width and depth of the micro-grooves, further investigation is necessary, such as the laser parameters for PEEK surface treatment and the direction of bonding with resin.

This study evaluated the SBS of PEEK to ICR or DBR using various surface treatment methods, and the LS group, which featured grid-like microslits created through laser engraving, demonstrated the highest SBS. Cohesive failure was observed in the AB, SA, and LS groups, whereas the NT group displayed adhesive failure. Resin residue was visible on the surfaces of the AB and LS groups, and resin penetrated the microslits of the LS group. These results suggest that laser engraving has the potential to enhance the bond strength and durability between PEEK and ICR or DBR, making it a promising surface treatment method for PEEK.

Declaration of competing interest

The authors have no conflicts of interest relevant to this article.

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