



# Shear bond strength of composite resin to high performance polymer PEKK according to surface treatments and bonding materials

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**PURPOSE.** The object of the present study was to evaluate the shear bonding strength of composite to PEKK by applying several methods of surface treatment associated with various bonding materials. **MATERIALS AND METHODS.** One hundred and fifty PEKK specimens were assigned randomly to fifteen groups (n = 10) with the combination of three different surface treatments (95% sulfuric acid etching, airborne abrasion with 50 µm alumina, and airborne abrasion with 110 µm silica-coating alumina) and five different bonding materials (Luxatemp Glaze & Bond, Visio.link, All-Bond Universal, Single Bond Universal, and Monobond Plus with Heliobond). After surface treatment, surface roughness and contact angles were examined. Topography modifications after surface treatment were assessed with scanning electron microscopy. Resin composite was mounted on each specimen and then subjected to shear bond strength (SBS) test. SBS data were analyzed statistically using two-way ANOVA, and post-hoc Tukey's test ( $P < .05$ ). **RESULTS.** Regardless of bonding materials, mechanical surface treatment groups yielded significantly higher shear bonding strength values than chemical surface treatment groups. Unlike other adhesives, MDP and silane containing self-etching universal adhesive (Single Bond Universal) showed an effective shear bonding strength regardless of surface treatment method. **CONCLUSION.** Mechanical surface treatment behaves better in terms of PEKK bonding. In addition, self-etching universal adhesive (Single Bond Universal) can be an alternative bonding material to PEKK irrespective of surface treatment method. [*J Adv Prosthodont 2017;9:350-7*]

**KEYWORDS:** Dental bonding; Shear bond strength; PolyEtherKetoneKetone; PEKK

## INTRODUCTION

PolyEtherKetoneKetone (PEKK), one of the high-performance polymers, was introduced to the dental field since it

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has a wide range of potential application. Also, PEEKs (polyetheretherketones) are presented as alternative materials to metal and glass ceramics,<sup>1</sup> because of their appropriate stress distribution, high fracture resistance, and low abrasion to the antagonist enamel. In medical area, PEEK, the well-known as one of the PAEK (polyaryletherketone) member, generally used as bio-implantation because of aforementioned characteristics and high biocompatibility.<sup>2</sup> It has received attention as alternative substances for the long-termly used titanium in orthopedic fields.<sup>3-5</sup> In dental area, the application of PEEKs as temporary abutment of dental implant is increasing.<sup>3,4,6</sup> Also, it has been used as dental clasps and frameworks for dental removable partial prostheses.<sup>7</sup>

Recently introduced PEKK, has about 80% higher compressive strength than that of PEEK according to the manufacturers' (Cendres+Métaux) reports.<sup>2</sup> A wider capability

of processing, including milling and pressing, makes PEKK attractive dental material for crowns and fixed dental prostheses (FDPs) as well as removable prostheses. In spite of its attractive mechanical properties and biocompatibility, the low translucency and the grayish color of PEKK are still limitation of its usage as a monolithic material for dental restoration.<sup>8</sup> Thus, to obtain satisfactory esthetics, additional veneering with adequate bonding to PEKK is prerequisite for its intraoral usage.

Until now, there are only few studies reporting bonding to the PAEK groups. Moreover, only one study was published for bonding to PEKK materials. According to the earlier study of assessing the tensile bond strength of differently surface treated PEEK,<sup>9</sup> in contrast to no bond obtained on the polished surface, adequate bond strength could be achieved on sulfuric acid (98%) etched, sandblasting with alumina, or silica-coating with Rocatec (3M ESPE) system. Another study demonstrated that the surface pre-treatment of PEEK with airborne abrasion system improved the adhesive properties of PEEK compared to acid-etching surface treatment groups.<sup>10</sup> According to the recent study which evaluated different primers on PEEK,<sup>11</sup> airborne abrasion surface treatment and priming with a multifunctional methacrylates containing primer (Luxatemp Glaze & Bond) could achieve a durable resin bond strength. Another study for bonding to PEKK materials,<sup>2</sup> evaluating the tensile bond strength with two different surface treatment and two different adhesive systems on PEKK, showed that the combination of silica-coating (Rocatec Soft), universal primer (Monobond Plus) and a resin primer (Luxatemp Glaze & Bond) achieved the highest and the most durable bonding ability. However, this study did not comparatively evaluate the surface characteristics such as topological chances, wettability and surface roughness of the specimen after surface treatment.

The bond strength can be achieved by surface treatment and some adhesive systems. However, most of the previous *in vitro* studies for bonding composite resin to high-performance polymer are limited to PEEK materials with few bonding materials. In addition, there are only few studies evaluating the effect of surface pre-treatment on the wettability and surface roughness of the PEKK surface. Therefore, the object of this study was to investigate the SBS of composite to PEKK materials with three different surface treatments, chemical and mechanical methods that are significantly effective to PEEK in the previous studies, and five bonding materials, MDP containing self-etching universal bonding materials as well as the bonding materials that were significantly effective to PEEK in the previous studies. Topology modification, the contact angle and surface roughness and of the specimen immediately after surface treatment were comparatively assessed. The null hypothesis was that surface treatment methods and bonding materials do not influence the SBS of composite resin to PEKK.

## MATERIALS AND METHODS

One hundred and fifty PEKK specimens with a dimension of  $7 \times 7 \times 2$  mm were obtained from a PEKK blank (Pektkon Ivory, lot no. 200368, Cendres+Métaux, Biel/Bienne, Switzerland). The PEKK substrates were embedded in polyester resin (Polycoat, Aekyung Chemical Co., Daejeon, Korea) with 20 mm diameter and 24 mm height (Fig. 2A, Fig. 2B). For surface standardization, the bonding surfaces in all specimens were polished with a series of rotating silicon carbide abrasive paper (220-grit up to 600-grit) under water rinsing. Before initiating surface treatment and bonding procedure, all specimens were cleaned ultrasonically for 10 minute in an ultrasonic bath containing distilled water and air-dried. The PEKK specimens were then assigned to

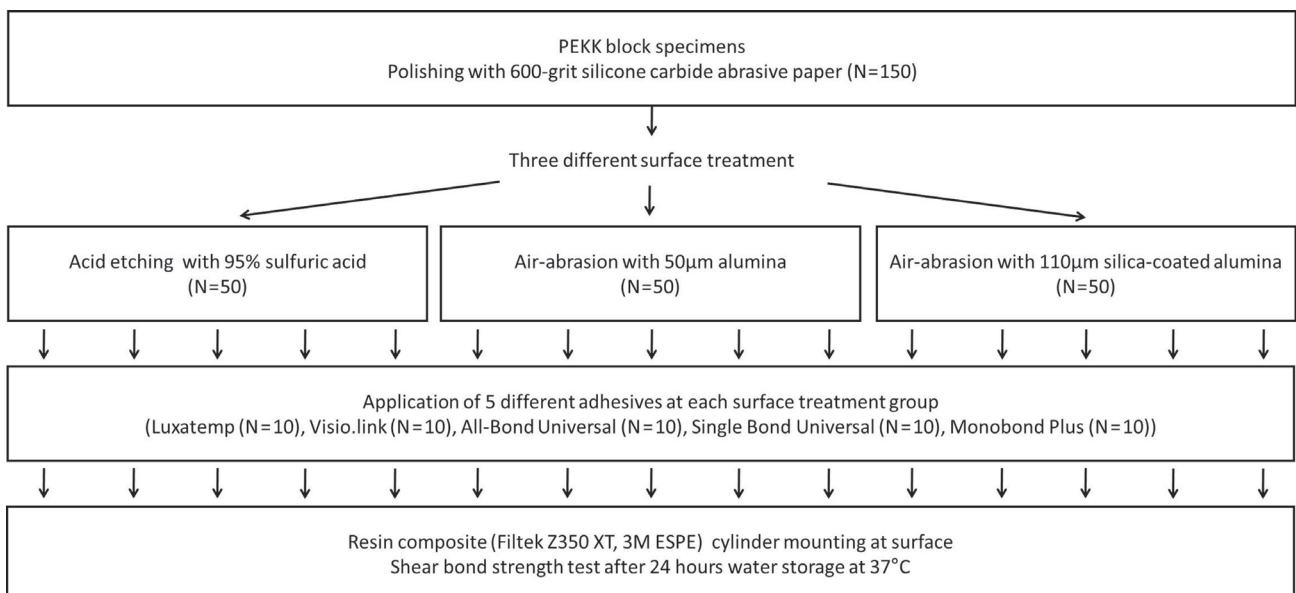


Fig. 1. Experiment design of this study.

three surface treatment groups, which received the followings (n = 50 for each surface treatment group, Fig. 1):

(A) Acid etching with 95% sulfuric acid (CAS: 7664-93-9, Duksan pure chemical Co., Ansan, Gyeonggi, Korea) for 1 minute. Careful rinsing with de-ionized water for 1 minute, then air-dried for 20 seconds.

(B) Air-abrasion with 50 μm alumina particle (LEMAT NT4, Wassermann, Germany) at 5 mm distance from the nozzle to the specimen for 20 seconds with a pressure of 0.5 MPa, then air-dried with compressed air for 20 seconds.

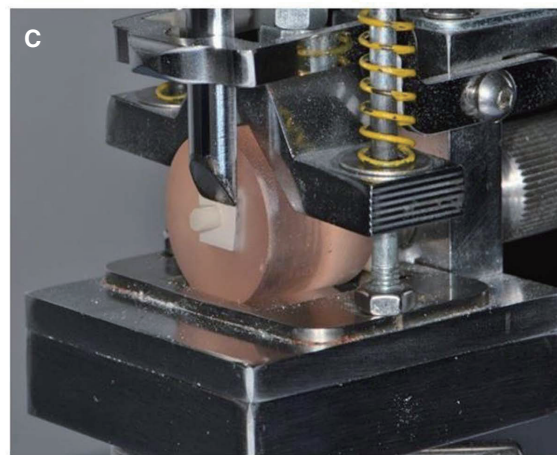
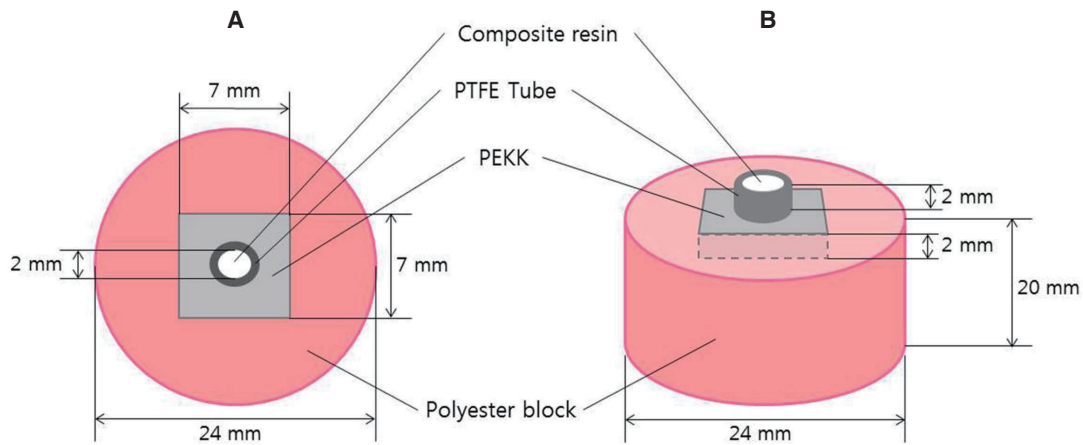
(C) Air-abrasion with 110 μm silica-coating alumina particle (Rocatec Plus, 3M ESPE AG, Seefeld, Germany) at 5 mm distance from the nozzle to the specimen for 20 seconds with a pressure of 0.5 MPa, then air-dried with compressed air for 20 seconds.

Two additional specimens of each surface treatment group were fabricated to examine the surface topography. After the respective surface treatment and sputter-coating with gold nanoparticles (thickness < 10 nm), these representative specimens were examined using a field emission

scanning electron microscope (FE-SEM; S-4800, Hitachi Co., Tokyo, Japan) under ×10,000 magnification.

Five additional specimens were fabricated of each surface treatment group to measure the wettability and surface roughness of each pre-treated surface. The wettability was examined by measuring the contact angle with a measurement machine (Phoenix 300 Touch, SEO, Suwon, Gyeonggi, Korea). Using an attached injector, 5 μL distilled water was dropped on the center of the specimen. Considering energy change with time, each image was taken and digitally recorded 10 seconds after injection. The contact angles were measured in the digital images with the measurement machine provided software. The measurements were performed repeatedly four times in each specimen. The values of surface roughness (Ra) of the specimens for each surface treatment group were examined using a surface profile-meter (TR200, TIME Group, Pittsburgh, PA, USA) with a cut-off value of 0.8 μm. The measurements were performed at four different points in each specimen.

Following the respective surface treatment, fifty samples



**Fig. 2.** Schematic drawings of specimen preparation for shear bond strength test. Top view (A) and three dimensional view (B) of resin mounted specimen. (C) Specimen in the shear bond testing machine.

of each surface pre-treatment group were divided randomly to one of the following five bonding procedures ( $n = 10$  for each bonding material group; Table 1). After a uniform thin layer of bonding material was applied onto the PEKK specimen, a thin PTFE (polytetrafluorethylene) tube (Sungjin, Seoul, Korea) with 3 mm inner diameter and 2 mm thickness was located at the center of each specimen (Fig. 2A, Fig. 2B). Resin composite (Filtek Z350 XT, 3M ESPE, St. Paul, MN, USA), generally used for bonding test in previous studies,<sup>12-15</sup> was filled in the tube and light-polymerized for 40 seconds, and again for 40 seconds after the PTFE tube had been cut and removed with a blade. Subsequently, all specimens were stored in 100% relative humidity at 37°C for 24 hours prior to bond strength test.

The SBS of composite resin to PEKK specimens was tested by using a universal test machine (AG-10KNX, Shimadzu Co., Kyoto, Japan). Load was applied using a knife-edge shaped with 1 mm/min crosshead speed (Fig. 2C). When the resin composite separated from the PEKK surface, the load at failure was measured. A shear bond strength was expressed in Mega-Pascals (MPa), calculating with dividing the maximum value of load at failure in newtons (N) by the bonding area in square millimeters. To evaluate each de-bonding aspect, the failure mode was assessed using a video inspection system (Optical video measuring system, Seven Ocean, Seoul, Korea) with  $\times 10$  magnification.

The SPSS statistical software version 22.0 for window (SPSS version 22, IBM, Armonk, NY, USA) was used for statistical analysis. The Shapiro-Wilk and Kolmogorov-Smirnov tests were performed to verify the normality of

contact angle, Ra and SBS data distribution. To evaluate the effect of surface pre-treatment method on wettability and Ra, one-way analysis of variance (ANOVA) and post-hoc Tukey's test was used. To examine the effect of different surface pre-treatments, bonding materials and interactions of the two factors on SBS, two-way analysis of variance (ANOVA) and post-hoc Tukey's test was used. All statistical analysis was performed with a significant level of 5%.

## RESULTS

SEM images of the differently pre-treated PEKK surfaces are shown in Fig. 3. While the surface of the only polished specimens showed a generally smooth surface with a little scratches and grooves (Fig. 3A), distinct surface modification were observed with all other pre-treated surfaces. In the sulfuric acid etching PEKK surface, numerous blister-like micro-porous were observed over the entire surface (Fig. 3B). The air-abrasion with the 50  $\mu\text{m}$  alumina and 110  $\mu\text{m}$  silica-coating alumina led the PEKK surfaces be more irregular, accentuated and dispersed surface pattern as compared to the chemically etched surface (Fig. 3C, Fig. 3D).

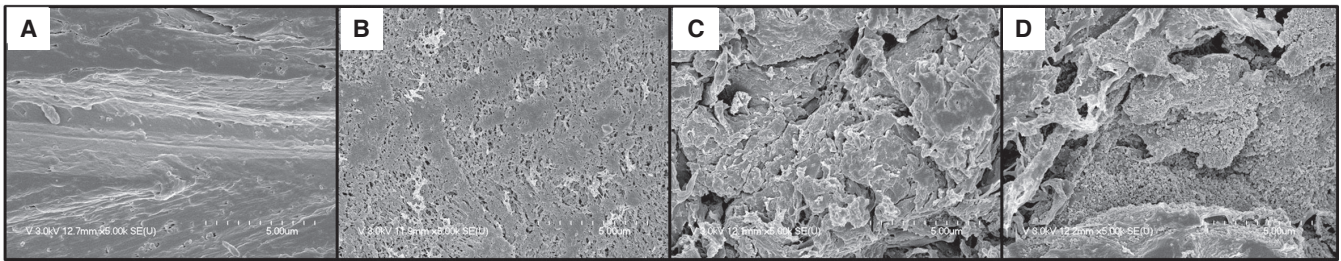
The representative contact-angle measurement images of each surface treatment group are illustrated in Fig. 4. Mean values ( $\pm$  SD) of contact angle and Ra are shown in Fig. 5. The contact angle of mechanical surface pre-treatment groups (AA and SA) showed statistically no significant difference between the two, and were significantly smaller than those of NT and SE ( $P < .05$ ). Regarding the surface roughness, Ra values of mechanical surface pre-treatment groups (AA and SA) showed statistically no significant dif-

**Table 1.** Bonding materials and procedures used in the present study

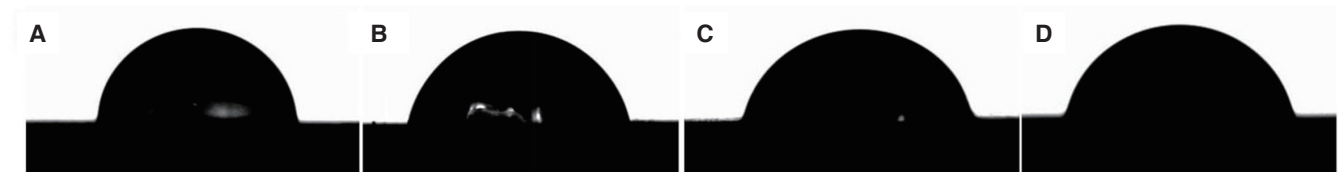
Product Name	Manufacturer	Batch No.	Composition	Application
Luxatemp Glaze & Bond	DMG, Hamburg, Germany	719757	Multifunctional acrylates, MMA, catalysts, stabilizers, additives	1) Apply a thin layer 2) Light cure 20 s
Visio.link	Bredent GmbH & Co KG, Senden, Germany	142655	MMA, PETIA, dimethacrylates, photoinitiators	1) Apply a thin layer 2) Light cure 90 s
Single Bond Universal	3M ESPE, Deutschland GmbH, Germany	497909	MDP phosphate monomer, dimethacrylate resins, HEMA, Vitrebond™ copolymer, filler, ethanol, water, initiators, silane	1) Apply a thin layer by rubbing for 20 s 2) Gentle air stream for 5 s 3) Light cure 10 s
All-Bond Universal	Bisco Inc, Schaumburg, IL, USA	1300000367	MDP, bis-GMA, HEMA, ethanol, water, initiators	1) Apply a thin layer by rubbing for 15 s 2) Gentle air stream for 10 s 3) Light cure 10 s
Monobond Plus + Heliobond	Ivoclar Vivadent AG, Schaan, Liechtenstein	T29123	Silane methacrylate, phosphoric acid methacrylate, sulfide methacrylate	1) Apply a thin layer of Monobond plus and vaporization for 60 s 2) Apply a thin layer of Heliobond and light cure for 10 s
		T34087	Bis-GMA, TEG-DMA	

MMA: methylmethacrylate; PETIA: pentaerythritol triacrylate; MDP: 10-methacryloyloxydecyl dihydrogen phosphate; HEMA: 2-hydroxyethyl methacrylate; Bis-GMA: bisphenol-A-diglycidylmethacrylate; TEGDMA: triethylene glycol dimethacrylate.

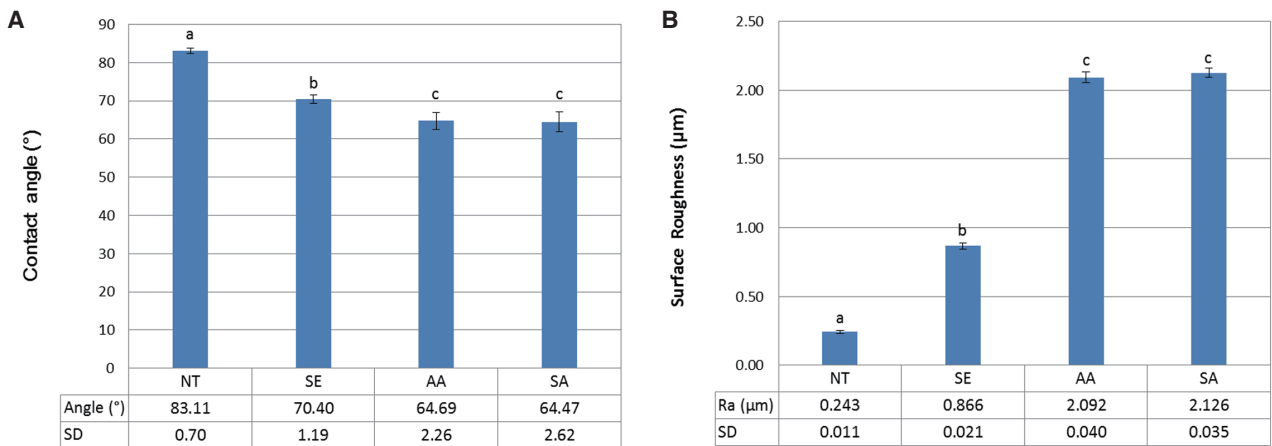




**Fig. 3.** SEM images of the different surface treatment at a magnification of 10,000×. (A) NT: non treatment; (B) SE: sulfuric etching; (C) AA: alumina particle abrasion; (D) SA: silica coated alumina particle abrasion.



**Fig. 4.** Typical images of contact angle for pre-treated surface specimen. (A) NT: non treatment; (B) SE: sulfuric etching; (C) AA: alumina particle abrasion; (D) SA: silica coated alumina particle abrasion. The contact angle of AA and SA specimens were significantly larger than those of NT and SE ( $P < .05$ ).

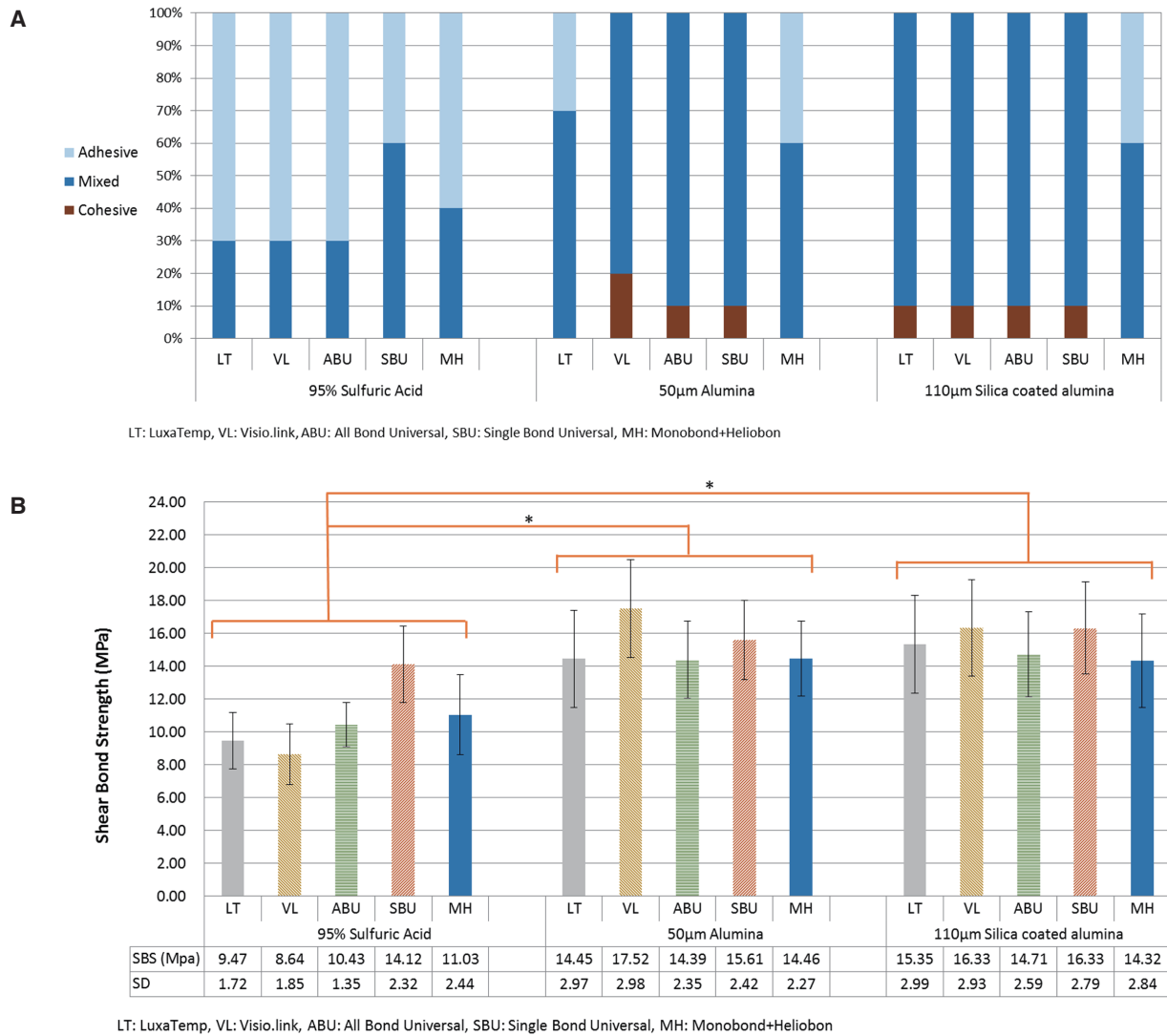


**Fig. 5.** Mean values ( $\pm$  SD) of contact angle (A) and surface roughness (B) after surface pre-treatment. Vertical bars mean that the standard deviation and identical letters indicate statistically significant difference between the experimental groups ( $P < .05$ ).

ference between the two, and were significantly greater than those of NT and SE ( $P < .05$ ).

Mean values of SBS and its standard deviations are illustrated in Fig. 6B. Two-way ANOVA indicated that the surface treatment and bonding materials significantly affected SBS ( $P = .004$ ). For the effects of the surface pre-treatment method, there was a significant difference of SBS values between mechanical surface pre-treatment groups (AA and SA) and chemical surface pre-treatment group (SE) regard-

less of bonding materials ( $P < .0001$ ). For the effects of the bonding materials, there was also a significant difference of SBS values between of Single Bond Universal (3M ESPE) groups and other bonding material groups regardless of surface treatment techniques. When evaluating with the combination effects of surface treatment techniques and bonding materials, all of the mechanically pre-treated groups with 50 μm alumina and Rocatec Plus (3M ESPE) and one of the chemically pre-treated groups, etched by sul-



**Fig. 6.** Failure mode of experimental groups (A) and mean values ( $\pm$  SD) of SBS (B). Asterisk (\*) indicates statistically significant difference between the experimental groups. Mechanical pre-treatment groups were significantly higher bonding strength than sulfuric acid treatment group. Single bond universal in sulfuric acid pre-treatment group showed no significantly different bonding strength from other mechanical treatment groups ( $P < .05$ ).

furic acid (95%) and Single Bond Universal (3M ESPE) applied, showed significantly higher SBS values, ranging from 14.12 to 17.52 MPa, than the other experimental groups, ranging from 8.64 to 11.03 MPa.

The failure mode of specimens showed three de-bonding types: 100% adhesive failure, 100% cohesive failure, and mixed failure. The failure mode distribution of de-bonded surface in this study is shown in Fig. 6A. In the chemical surface pre-treatment group (SE), no cohesive failure mode was observed and adhesive failure mode was dominant. In the mechanical surface pre-treatment groups (AA and SA), mixed failure mode was predominantly observed.

## DISCUSSION

This study evaluated the effect of the different surface pre-treatment methods and various bonding materials on SBS of composite resin to PEKK. The results of this experiment showed that there was a significant improvement in SBS of PEKK using the air-abrasion techniques in comparison with sulfuric acid surface treatment. In addition, one bonding material showed the consistent bonding strength irrespective of the type of surface treatment. Therefore, the null hypothesis that there is no significant difference of effects on SBS between surface treatment techniques and between bonding materials has to be rejected.

The present study used a sulfuric acid for the chemical surface treatment and air-abrasion techniques for the mechanical surface treatment on PEKK. According to the earlier study, the functional carbonyl and ether groups between the benzene rings are attacked by sulfuric acid.<sup>10</sup> This chemical reaction makes more functional groups be exposed to bond to components of the adhesive material. As a result, the increasing of surface polarity and the enhancement of the adhesive materials diffusion into PEEK polymer can make the bond strengths be higher.<sup>10</sup> Another previous study, when sulfuric acid is used, a sulfonating of the benzene ring in PEEK molecule can be achieved theoretically.<sup>16</sup> According to the other studies investigating the bond strength of differently surface pre-treated PEEK, when comparing the surface treatment with only sulfuric acid, although abrasion with airborne particle results in an improvement micro-roughness, bond strength was higher in sulfuric acid pre-treatment group.<sup>17</sup> As the result of that study, the importance of chemical linking on the polymer was emphasized. On the basis of the SEM images (Fig. 3) and result of Ra and contact angle measurement (Fig. 5), the present study also showed the similar surface roughness improvement in air-abrasion pre-treatment groups. However, in contrast to the previous study,<sup>17</sup> bond strength was higher in air-abrasion treatment groups. A possible explanation for this difference is that the air-abrasion treatment more effectively enhancing the surface roughness and wettability in PEKK than PEEK to allow better mechanical retention. Further studies comparatively investigating the effect of surface treatment on PEKK and PEEK are therefore required.

The chemical compositions of the bonding materials are also important in bonding to the polymers. The results from the present study showed that there was a significantly different effect on SBS of PEKK between the bonding materials. As previously mentioned, all of the sulfuric acid etching pre-treated groups showed lower SBS values than air-abrasion surface treatment groups regardless of bonding materials except Single Bond Universal (3M ESPE) employed groups. In other words, Single Bond Universal (3M ESPE) employed experimental groups showed no significantly different SBS values according to the surface treatment techniques.

To the authors knowledge, this study firstly used the MDP-containing bonding materials - All-Bond Universal (BISCO) and Single Bond Universal (3M ESPE) - as well as MMA-containing adhesive systems - Luxatemp Glaze & Bond (DMG), Visio.link (Bredent), Monobond Plus (Ivoclar vivadent), and Heliobond (Ivoclar vivadent) - to examine SBS of resin composite to PEKK. Kern and Lehmann<sup>11</sup> evaluated the TBS of composite resin to PEEK with different surface pre-treatment techniques and bonding materials and observed the highest TBS in the specimens of using MMA-containing adhesive material. Stawarczyk *et al.*<sup>8</sup> also evaluated the TBS of veneering composite resins to PEEK with the comparing different bonding materials and reconfirmed these findings, reporting that MMA-containing

bonding materials demonstrated the highest bond strength. However, in this study, MDP-containing adhesive materials, both of All-Bond Universal (BISCO) and Single Bond Universal (3M ESPE), also showed no significantly different SBS values from that of MMA-containing bond materials. This may be attributed to the fact that MDP has similar effect to MMA-containing bond materials on roughened PEKK surface. As having a hydrophobic methacrylate terminal end and a hydrophilic phosphate terminal end, copolymerizing resin monomers and chemically binds to oxides, respectively, MDP has a bifunctional adhesive monomer that can bind to zirconia or metal.<sup>18-22</sup>

In the group of sulfuric acid etched surface, Single Bond Universal (3M ESPE) applied group showed significantly higher SBS values than All-bond universal applied groups, although the same phosphate monomer was included in both products. The main different composition between two universal bonding materials is silane, which is only present in Single Bond Universal (3M ESPE). Silane is adhesive promoters that have two different reactive functional groups which react and couple with various organic and inorganic materials. It is used to increase the union between dissimilar materials. The hydrolysable functional groups can react to the surface of hydroxyl groups of inorganic substrates generating a siloxane bond (Si-O-Si). The non-hydrolysable organic functional group, having a carbon-carbon double bond, can polymerize with the monomers of composite resin.<sup>23,24</sup> Although it could be assumed that there might be some amount of the hydroxyl groups of PEKK substrates exposed after sulfuric acid surface treatment, more investigations are needed.

The present study evaluated the effect of surface pre-treatment techniques and various bonding materials on SBS of composite resin to PEKK and revealed that the combination of air-abrasion surface treatment techniques and not only methacrylate containing adhesive systems but also MDP containing universal bond materials results in suitable bond strengths. However, to investigate the long-term success of veneering resins with PEKK materials, additional studies considering the oral environment and aging effects are required.

## CONCLUSION

When bonding resin composite to PEKK materials, the combination of air-abrasion surface treatment with MDP or MMA-containing bond materials are recommended and acid surface treatment of PEKK is not required. However, regardless of surface treatment method, silane containing self-etching universal adhesive (Single Bond Universal) can be an effective bonding material to PEKK.

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