



Original Article

# Influence of handheld nonthermal plasma on shear bond strength of polyaryletherketone to resin-matrix cement



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## KEYWORDS

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Superhydrophilic;  
Shear bond strength;  
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stress aging tests;  
Resin-matrix cement

**Abstract** *Background/purpose:* Challenges exist regarding the bonding efficiency of polyaryletherketone (PAEK), a high-performance thermoplastic, attributed to its chemical inertness and hydrophobic surface, hindering effective bonding with resin-matrix cement. This research explored the impact of handheld nonthermal plasma (HNP), under varying operational parameters, on PAEK surface wettability and changes in bonding performance with cement.

*Materials and methods:* Three types of disc-shaped PEAK specimens were prepared, with surface treatments categorized as grinding, airborne-particle abrasion (APB), and HNP. Surface wettability was analyzed using a contact angle analyzer ( $n = 10$ ). Specimens were bonded with resin cement and subjected to artificial aging tests: distilled water bath (NA), thermocycling, and highly accelerated stress tests ( $n = 10$  for each test). Shear bond strength (SBS) was measured, failure modes were analyzed, and statistical analyses were conducted.

*Results:* The HNP markedly improved PAEK surface wettability, achieving superhydrophilicity ( $P < 0.05$ ). This effect intensified with extended operation times (30 or 60 s) and reduced elapsed times ( $<30$  s). HNP-treated PAEK exhibited higher SBS than APB ( $P < 0.05$ ) and maintained bonding durability after artificial aging, particularly in ketone-enriched variants. Failure analysis revealed predominantly adhesive failure under APB–NA treatment, mixture failures under HNP–NA treatment and postaging, but no cohesive failure.

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**Conclusion:** The HNP device benefits dental settings by transforming the PAEK surface into superhydrophilic properties, thereby improving PAEK–cement bonding. It significantly enhances bond durability within 30 s of operation and after a 30 s elapsed period. It is noteworthy that ketone-enriched PAEK demonstrates markedly improved bonding performance. © 2024 Association for Dental Sciences of the Republic of China. Publishing services by Elsevier B.V. This is an open access article under the CC BY-NC-ND license (<http://creativecommons.org/licenses/by-nc-nd/4.0/>).

## Introduction

Polyaryletherketone (PAEK), a semicrystalline, high-performance thermoplastic polymer, is synthesized by linking aromatic phenyl with varying ether or ketone group ratios. Prominent variants include polyetheretherketone (PEEK) and polyetherketoneketone (PEKK), recognized in dentistry for their bone-like mechanical properties, providing an alternative to traditional metal or zirconia frameworks.<sup>1–4</sup> PAEK, with its inherent low translucency and opaque gray shade, is commonly modified with pigment fillers for color adjustment. Additionally, the incorporation of carbon fibers, nanoceramic fillers, or metal oxides enhances its mechanical properties for dental applications.<sup>4–7</sup> Challenges arise from the chemical inertness and low surface energy of PAEK, impeding effective bonding with resin-matrix cements, which has become a focus of contemporary research.<sup>8,9</sup> Surface modifications of PAEK, including mechanical treatments (air abrasion, lasers, or grinding) and chemical treatment (etching with caustic solutions or applying bonding primers), are crucial for enhancing its workability and bonding efficacy.<sup>3,7,10,11</sup> Knowledge concerning that air abrasion or caustic solution etching combined with a bonding primer is deemed most effective, clinical application challenges exist, including human error during air abrasion and risks associated with caustic solution etching, necessitating caution.<sup>4</sup>

Recent advancements in dental clinics include the adoption of clinic-friendly handheld plasma devices. Plasma treatment, inducing functional groups to enhance hydrophilicity and bond strength, is increasingly favored in dentistry.<sup>1,6,9,12,13</sup> Previous studies have highlighted the effectiveness of cold plasma, particularly combined with acetylene gas, in bolstering the bond between PEKK and veneering composites.<sup>14</sup> Similarly, atmospheric-pressure plasma treatment enhances the bond strengths of PEEK owing to chemical bonding at the interface.<sup>8</sup> Researchers have found that different feeding gases for plasma treatment, such as hydrogen, nitrogen, helium, or argon, do not significantly affect bond strength.<sup>5,6,9,13</sup> However, conventional plasma methods often require high power or temperature, limiting chair-side use.<sup>5,13,14</sup> In contrast, handheld plasma devices overcome these limitations, enabling safe chair-side implant treatments that support efficient healing and expedite tissue regeneration or osseointegration.<sup>13,15</sup> Nevertheless, with a power output of only 18 W and a frequency range of 30–50 kHz, the efficacy of handheld plasma in altering the hydrophobic surface of PAEK materials warrants further investigation. Additionally, a research gap

exists regarding the bonding performance between PAEK materials with varying ether–ketone ratios or microfillers and cements following nonthermal plasma treatment.

The purpose of this study was to investigate the impact of handheld nonthermal plasma, under varying operational parameters, on the wettability of PAEK surfaces and changes in bonding performance between treated PAEK and cements. Two null hypotheses were posited (1) plasma treatment does not alter the surface wettability and bonding performance of PAEK, and (2) the elapsed time postplasma treatment does not affect surface wettability.

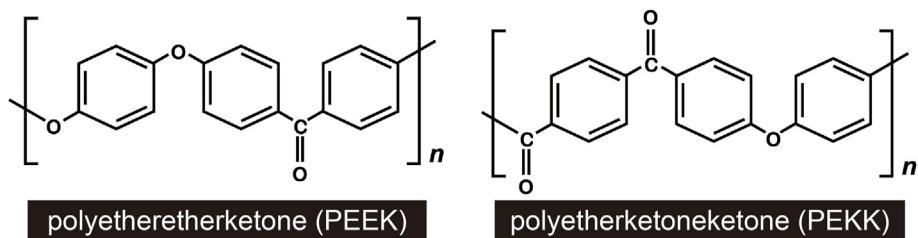
## Materials and methods

### Sample fabrication and surface treatment

The materials used in this study are outlined in Fig. 1 and Table 1. Disc-shaped specimens (Ø10 mm; 2.5 mm thick) of BP (BioHPP), VK (VESTAKEEP), and PK (pekkton) were designed using SOLIDWORKS 2013 software (Dassault Systèmes SolidWorks Corporation, Waltham, MA, USA) and milled using a Zirkonzahn dental CAD/CAM system (Milling Unit M1, Zirkonzahn GmbH, Gais, Italy). Postmilling, specimens underwent grinding, ultrasonic cleaning, and drying at 37 °C. The following surface treatments were applied: CON, no further treatment; APB, 10 s airborne-particle abrasion at 2 bars; and HNP, nonthermal plasma treatment with different operation times (O<sub>T</sub>10, O<sub>T</sub>30, or O<sub>T</sub>60) and elapsed times (0 s, 30 s, 60 s, 5 min, 10 min, or 30 min) using a handheld plasma unit (PiezoBrush PZ3, relyon Plasma GmbH, Regensburg, Germany).

### Surface wettability analysis and bonding procedure

Contact angles (CAs) were measured using a contact angle analyzer (Phoenix Mini, Surface Electro Optics Co., Ltd., Kunpo, South Korea) by dispensing 50 µL droplets to evaluate wettability in each treatment group (n = 10). A clear acrylic ring with a 5 mm diameter was positioned on each specimen. Resin cement (G-CEM LinkAce, GC Corp., Tokyo, Japan) was injected into the ring, followed by placement of a custom cap with a 4.9 N load for uniform bonding. Excess cement was removed, and light curing was performed following manufacturer guidelines. Subsequently, specimens were oven-dried at room temperature for 60 min for complete setting, followed by random allocation to three artificial aging groups (all n = 10): NA, 24 h in a 37 °C

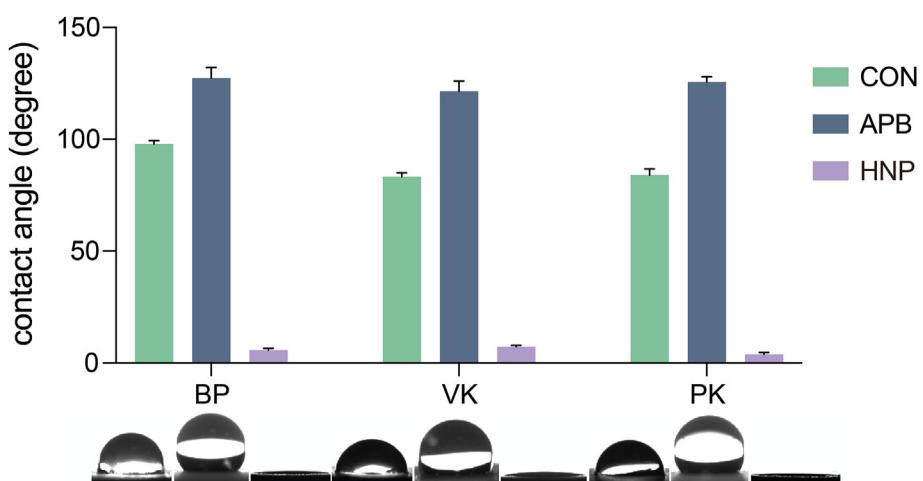


**Figure 1** Two materials within the polyaryletherketone (PAEK) family: polyetheretherketone (PEEK) and polyetherketoneketone (PEKK).

**Table 1** Materials tested in the study.

Trade Name	Main composition	Manufacturer	Lot number	Abb.
breCAM BioHPP	PEEK nanoceramic filler	Bredent, GmbH, Germany	496296	BP
VESTAKEEP (DC4450 R)	PEEK titanium dioxide pigment	Polyplastics-Evonik Corporation Ltd., Japan	57781699	VK
Pekkton ivory	PEKK titanium dioxide	Cendres + Métaux SA, Switzerland	389520	PK
Cobra (110 µm)	Al <sub>2</sub> O <sub>3</sub> , SiO <sub>2</sub> , etc.	Renfert GmbH, Germany	2327409	—
G-CEM LinkAce	Paste A: bis-GMA, UDMA, dimethacrylate, etc. Paste B: bis-MEPP, UDMA, dimethacrylate, etc.	GC Corp., Japan	022009	—

Abb., Abbreviation; PEEK, polyetheretherketone; PEKK, polyetherketoneketone; UDMA, urethane dimethacrylate; MMA, methylmethacrylate; bis-GMA, bisphenol A diglycidylether methacrylate; bis-MEPP, bisphenol A ethoxylate dimethacrylate.



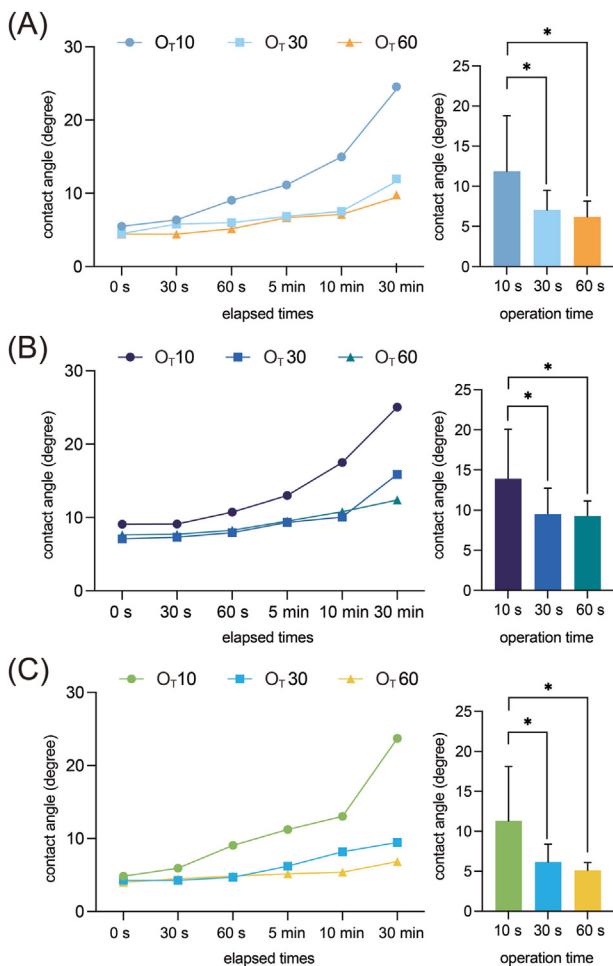
**Figure 2** Contact angle analysis results for different surface treatments. CON: surface polished with #600 silicon carbide abrasive paper; APB: surface abraded with 110 µm alumina particles under 2 bar pressure for 10 s; HNP: surface subjected to nonthermal plasma treatment for 30 s (O<sub>T</sub>30), followed by an elapsed period of 30 s.

distilled water bath; TC, 10,000 cycles of thermocycling aging at 5°C–55 °C, as per ISO 10477; HA, highly-accelerated stress aging at 134 °C and 2 bar for 5 h, as per ISO 13356.

#### Shear bond strength (SBS) test

The SBS was measured using a universal testing machine (JSV-H1000, Japan Instrumentation System, Nara, Japan) at

a crosshead speed of 1 mm/min until failure at the bonded interface. Following SBS testing, debonded specimen interfaces were analyzed using a dental microscope. Failure modes were categorized using the adhesive remnant index (ARI),<sup>16</sup> based on the percentage of residual cement on the PAEK surface: score 0 (0%: adhesive failure), scores 1 and 2 (<50% and >50%, respectively: mixture failure), and score 3 (100%: cohesive failure). Representative debonding interfaces were recorded using a stereo microscope (Leica M205C, Leica Microsystems GmbH, Darmstadt, Germany)



**Figure 3** Contact angle analysis results under various plasma conditions. Contact angle analysis results for samples derived from different PAEK materials under various plasma conditions. (A), (B), and (C) represent BP, VK, and PK, respectively. Plasma conditions include six elapsed times (0 s, 30 s, 60 s, 5 min, 10 min, and 30 min) and three operation times: 10 s ( $O_T10$ ), 30 s ( $O_T30$ ), and 60 s ( $O_T60$ ). Asterisks (\*) indicate a statistically significant difference between groups ( $P < 0.05$ ).

equipped with an automated focus-stacking microscopy system (TORI FOCUS, Taiwan Ocean Research Institute, Kaohsiung, Taiwan) at  $\times 4$  and  $\times 20$  magnifications.

### Statistical analysis

Data, represented as mean and standard deviation values, were statistically analyzed using SPSS (v19; IBM Corp, Armonk, NY, USA) and Prism (v10; GraphPad Software Inc.; San Diego, CA, USA), with the significance level set at 5%. Normality distribution was evaluated using Shapiro–Wilk tests. One-way analysis of variance was used to assess differences in the SBS of PAEK after surface treatments and artificial aging, with the Tukey honestly significant difference test employed for group comparisons.

## Results

### Surface characteristics

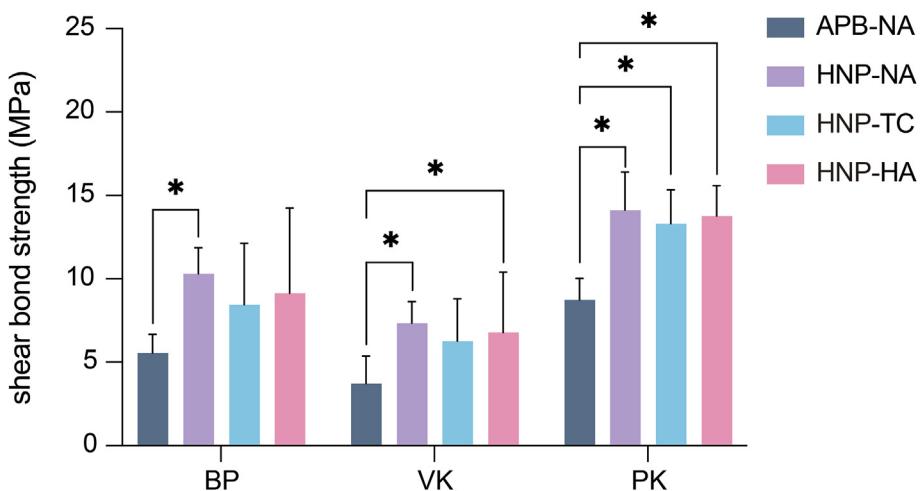
**Fig. 2** illustrates the changes in PAEK surface wettability following various surface treatments. The results revealed substantial variations among different surface treatments ( $P < 0.05$ ). For instance, CON exhibited a hydrophobic surface ( $CA > 83.35^\circ$ ), whereas APB transformed the surface into a superhydrophobic state ( $CA > 121.55^\circ$ ). In contrast, HNP-treated surfaces transitioned to a superhydrophilic state ( $CA < 7.32^\circ$ ), particularly when HNP was applied with an  $O_T$  of 30 s and an elapsed time of 30 s. The impact of different plasma conditions on PAEK surface wettability is presented in **Fig. 3**. Although  $O_T10$  improved hydrophilicity, its surface continued to exhibit substantial hydrophobicity compared with  $O_T30$  and  $O_T60$ . No significant differences in hydrophilicity improvement were observed between  $O_T30$  and  $O_T60$  ( $P > 0.959$ ). Elapsed times of 0 and 30 s did not lead to significant differences in surface wettability ( $P > 0.05$ ), with superhydrophilic characteristics ( $CA < 10.73^\circ$ ) emerging under these periods. However, after a 30 min elapsed period, the PAEK surface's hydrophilicity was significantly reduced ( $P < 0.05$ ).

### Bonding performance

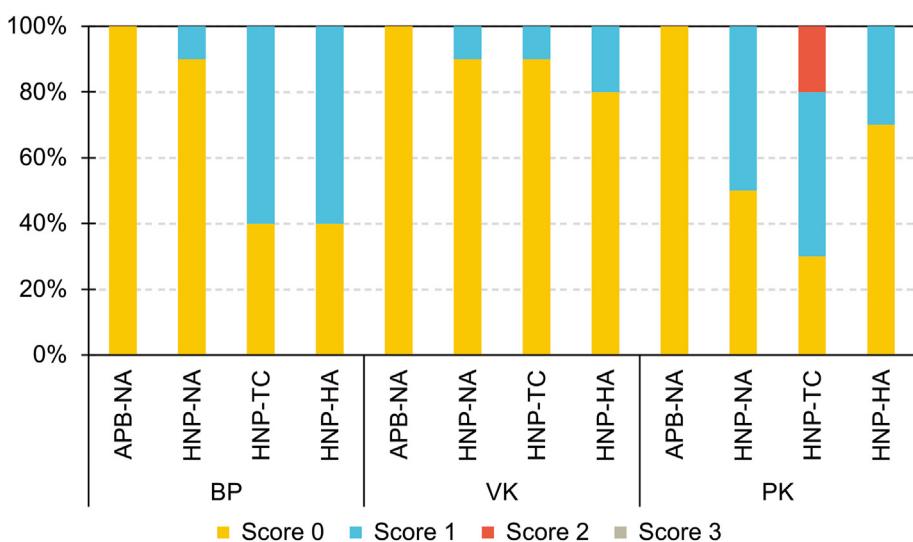
SBS results for PAEK following various surface treatments with cement are depicted in **Fig. 4**. Prior to artificial aging tests, HNP demonstrated significantly higher SBS compared with APB, irrespective of treatment ( $P < 0.05$ ). Postaging (HNP–TC and HNP–HA), all materials exhibited a slight decrease in SBS compared with HNP–NA ( $P > 0.05$ ) yet their SBS remained significantly higher than that of APB–NA ( $P < 0.05$ ). Under various conditions, PK consistently exhibited the highest SBS ( $P < 0.05$ ) and was less affected by artificial aging, with reduction rates of 5.74% for TC, 2.62% for HA, and  $>10\%$  for BP and VK. Debonding fracture modes based on ARI are shown in **Fig. 5**, with microoptical images of debonding fracture surfaces presented in **Fig. 6**. Prior to artificial aging tests, APB–NA predominantly exhibited score 0; however,  $>90\%$  of HNP–NA showed score 0, indicating adhesive failure. Postaging, there was an increase in the frequency of score 1. Optical images revealed minor cement residues in VK (**Fig. 6A**). Cement residues were larger in BP but remained at  $< 50\%$ ; therefore, they were given score 1 (**Fig. 6B**), indicating mixture failure. Notably, 20% of PK in the HNP–TC group received score 2, with significant ( $>50\%$ ) cement residue observed (**Fig. 6C**). Cohesive failure (score 3) was not observed in this study.

### Discussion

In this study, the impact of HNP on modifying the PAEK surface structure and its bonding efficacy with cement was assessed. Results confirmed that HNP significantly enhances PAEK surface wettability, concurrently improving bonding performance, thereby refuting the initial null hypothesis. Air abrasion with alumina particles, considered the gold standard



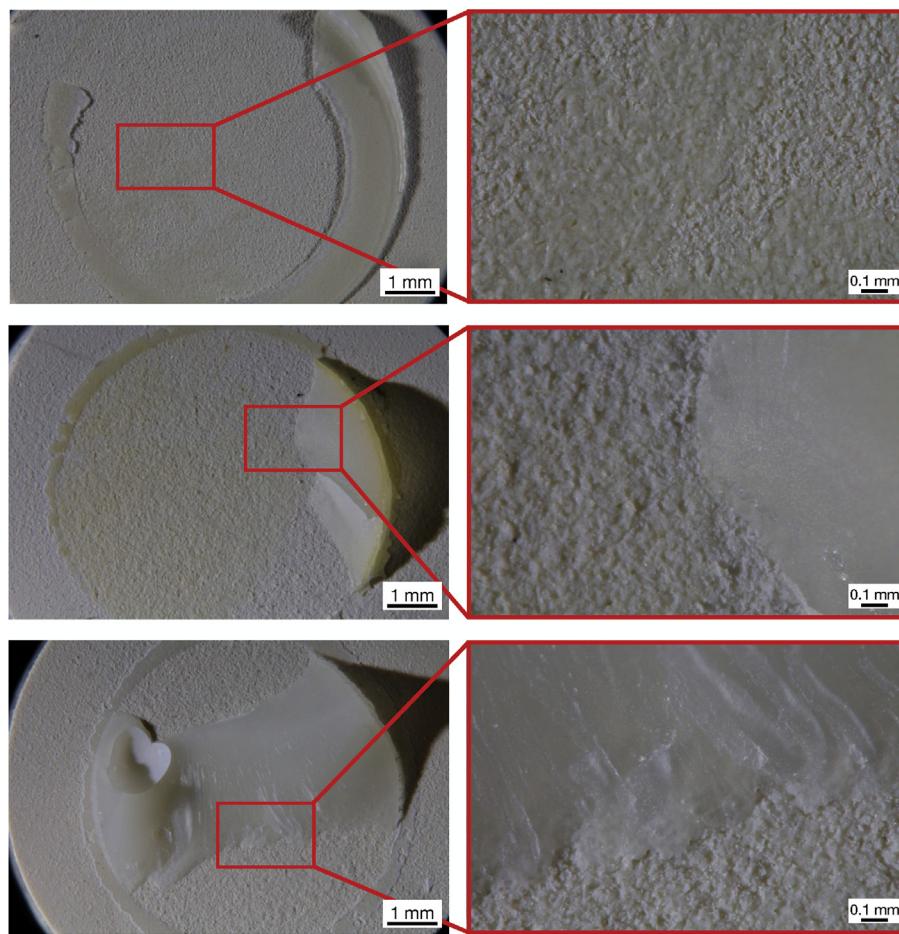
**Figure 4** Shear bond strength (SBS) test results. Asterisks (\*) indicate a statistically significant difference between groups ( $P < 0.05$ ).



**Figure 5** Failure mode evaluation. Evaluation of failure modes based on the adhesive remnant index. APB, airborne-particle abrasion treatment; HNP, nonthermal plasma treatment; NA, without artificial aging; TC, after thermocycling aging; HA, after highly-accelerated stress aging. Score 0: absence of resin-matrix cement on the PAEK surface; score 1: <50% coverage of resin-matrix cement; score 2: >50% coverage; score 3: full coverage of the PAEK surface (100%) by resin-matrix cement (not observed in this study).

method for surface preparation in dentistry, increases surface roughness, enlarging the contact area between PAEK and cement to improve bonding. Studies have shown that air abrasion accumulates aluminum and reduces oxygen and carbon content on the PAEK surface, improving bonding effectiveness.<sup>6</sup> However, potential drawbacks exist, such as operator skill dependence.<sup>2</sup> Given PAEK's robust chemical resistance, researchers have explored physicochemical methods, including caustic solution application for etching. This process involves sulfonation of PAEK with  $H_2SO_4$ , generating polar  $SO_3H$  groups, which facilitate interaction and rearrangement of PAEK molecular chains, thereby increasing bonding sites with resin.<sup>9,11</sup> However, this method can be hazardous and impractical for dental settings.<sup>3</sup>

Plasma can effectively process complex structures with a minimal depth of 10 nm, ensuring that overall material performance remains unchanged, offering a safe alternative for PAEK surface modification.<sup>6,9,12–14</sup> Although the outcomes of plasma treatment in improving SBS may vary among research teams, it is generally accepted that plasma forms new functional groups on PAEK's surface through a chemical bonding mechanism, thereby increasing surface free energy (SFE) and wettability.<sup>1,6</sup> Previous studies predominantly employed high-power systems for cold plasma or low-pressure, with conditions such as 0.3 mbar, 40–100 kHz, and 100–200 W, which are impractical for routine dental practice.<sup>5,13,14</sup> Conversely, in this study, a more practical, jet-type plasma device (PiezoBrush PZ3,



**Figure 6** Fracture intersurface morphology of representative samples after debonding. (A) Debonded intersurface of VK not subjected to aging tests, showing an adhesive remnant index (ARI) score of 1. (B) Debonded intersurface of BP following a highly accelerated stress test, showing an ARI score of 1. (C) Debonded intersurface of PK after a thermal cycle test, showing an ARI score of 2.

relyon Plasma GmbH) was employed, which is suitable for use in dental settings.

Hydrophilic surfaces are known to benefit cement in PAEK applications. Untreated PAEK typically exhibits hydrophobic surfaces, with contact angles varying based on the filler content in PAEK and averaging  $70^{\circ}$ – $90^{\circ}$ .<sup>5,7</sup> The current study aligns with previous findings (CON; Fig. 2), with nanoceramic fillers of BP introducing significant hydrophobicity ( $CA = 97.89^{\circ}$ ).<sup>17</sup> Air abrasion can leave alumina residues on PAEK surfaces, creating superhydrophobicity ( $CA > 120^{\circ}$ ), a finding consistent with these results (Fig. 2).<sup>7</sup> Following air abrasion, HNP effectively removes alumina and organic residues from PAEK surfaces, introducing  $-COOH$  groups and substantially reducing contact angles.<sup>6</sup> In the present study, HNP-treated specimens exhibited a significant reduction in average contact angle (Fig. 2), from  $124.88^{\circ}$  (APB) to  $5.68^{\circ}$  (HNP), confirming surface activation posttreatment, with increased functional groups and enhanced surface polarity, resulting in superhydrophilicity ( $CA < 10^{\circ}$ ) and markedly impacting SBS effectiveness (Fig. 4).<sup>14</sup>

Plasma has been shown to increase the SFE and wettability of PAEK; however, the effects of elapsed time

following plasma treatment on PAEK's surface characteristics remained unclear.<sup>1,5,6,9,12–14</sup> The present study demonstrated that the wettability of test specimens progressively decreases from 0 s to 30 min following HNP, with no significant difference observed within the first 30 s, refuting the second null hypothesis (Fig. 3). Regarding operation time, a previous study recommended 60 s, but the authors found that  $O_{T10}$  is insufficient for creating superhydrophilic surfaces ( $CA > 10^{\circ}$ ), with an operation time  $>30$  s being required.<sup>12</sup> Consequently, within the constraints of this study, a combination of  $O_{T30}$  and a 30 s elapsed time is proposed as the optimal HNP parameter (Fig. 3).

HNP led to a significant increase in SBS from 5.98 MPa (APB–NA) to 10.76 MPa (HNP–NA), attributed to increased SFE and the formation of functional groups on PAEK's surface (Fig. 4).<sup>5,6</sup> Plasma induces hydroxyl and carboxylic acid group formation on PAEK, altering its originally nonpolar surface characteristics. It also disrupts C–C and C–H bonds in the PAEK chain, generating free radicals, and providing additional bonding sites for chain transfer reactions, thereby allowing cement to penetrate PAEK surface pores and establishing effective bonding. Previous

studies have indicated a reduction in SBS between PAEK and cement due to thermocycling aging, primarily caused by thermal expansion leading to water molecule infiltration at the adhesive interface, reflecting the weak bond between PAEK and cement.<sup>7</sup> In the present study, specimens were subjected to TC, simulating one year of hot and cold exposure in an oral environment, as per ISO 10477, or HA at 134 °C and 2 bar for 5 h in a 65% humidity environment, equivalent to 10 years in a moist oral environment, as per ISO 13356. Results indicated a decreasing trend in SBS for HNP-treated specimens, although the reduction was not statistically significant. This suggests that HNP, inducing surface modification through chemical bonding, facilitates robust PAEK–cement bonding, achieving the desired bonding durability.

BP comprised 0.3–0.5 μm nanoceramic fillers in a PEEK matrix, whereas VK and PK contained approximately 20% TiO<sub>2</sub> powder in PEEK and PEKK matrices, respectively. Fillers and pigments in PAEK are known to impact SBS.<sup>6</sup> Added pigments significantly influence VK, imparting dental shade and pigment distribution on the material surface may contribute to the lower SBS observed in VK, warranting further investigation.<sup>5</sup> PK consistently exhibited higher SBS under all surface treatments. HNP creates functional groups in PAEK by breaking C–O–C and C=O–O bonds. Given the enrichment of ketone groups in PEKK, HNP generates additional binding sites for cement. Additionally, high-energy particles from HNP remove surface contaminants and reduce hydrophobic groups (–CH<sub>2</sub>) while increasing oxygen-containing groups (C=O). They break down macromolecular chains and disrupt C–H and C–C bonds, enhancing surface cleanliness and hydrophilicity and thereby improving bonding effectiveness.<sup>8,14</sup>

To limit variables, this study excluded bonding primers for chemical treatment. While combining HNP with a bonding primer may enhance SBS, this omission is a limitation. Future research should include bonding primers to understand better their interaction with HNP and their effect on bonding performance with cement. Additionally, further exploration is needed on the influence of biological factors (oral environment conditions), molecular-level changes in PAEK materials induced by HNP, and the impact of different types of cement on bonding performance.

The findings of this *in vitro* study emphasize that PAEK, particularly ketone-enriched PAEK, exhibits markedly better bonding performance. Using a handheld plasma device was found to improve surface wettability substantially, transforming the surface to a superhydrophilic state, thereby improving the PAEK–resin-matrix cement bond. Furthermore, the influence of chemical bonding mechanisms was notably evident with a 30 s operation followed by a 30 s elapsed period, resulting in enhanced bond durability, which persisted even under the challenges of artificial aging conditions.

## Declaration of competing interest

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

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