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# Influence of low-temperature atmospheric pressure plasma treatment on the surface treatment of fiber post

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**Low-temperature atmospheric pressure plasma (LT-APP) treatment can be used for surface energy activation and contaminant removal from minute gaps between fiber posts without increased risk of deformation due to physical force or heat. It is an effective method of surface treatment in clinical dentistry. We investigated its efficacy for fiber posts. In Experiment 1, we evaluated the impact of LT-APP on the bending strength of the fiber posts. A three-point bending test was conducted using a universal testing machine. In Experiment 2, we investigated the effect of LT-APP treatment on the adhesion strength between the fiber post and both the composite resin core and the adhesive resin cement. A cupping test was conducted using a universal testing machine.**

**Experiment 1 showed that the LT-APP treatment did not weaken the physical properties of the fiber post. Experiment 2 showed that when the LT-APP treatment was performed before the silane treatment, the bond strength improved between the fiber post, and both the composite resin core and the adhesive resin cement. The results indicated that LT-APP treatment of fiber posts is effective. (J Osaka Dent Univ 2019 ; 53 : 39-47)**

**Key words : Low-temperature atmospheric pressure plasma ; Fiber post ; Composite resin core ; Adhesive resin cement**

## INTRODUCTION

Crown dentin loss caused by caries or restorative procedures frequently occurs in teeth planned for endodontic treatment, resulting in the need for abutment construction. Metal posts are conventionally used for abutment construction. However, it has been reported that differences between the elastic modulus of metal posts composed of gold−silver−palladium alloy (8.8 kgf/mm$^2$) and that of dentin (1.4 kgf/mm$^2$) increase the risk of tooth root fractures.[1-4] On the other hand, fiber posts have a bend elastic constant similar to that of dentin and thus produce less stress concentration on the tooth root compared to metal posts.[5-7] Moreover, because root fractures occur near the tooth cervix, tooth extraction can often be avoided.[8-10]

Fiber posts are frequently used to treat patients with metal allergies to prevent gingival discoloration because of the elution of metal ions, and to repair cosmetic dental prostheses. However, although fiber posts offer several advantages, fiber-post adhesion with cement is reportedly more difficult than that with other materials.[11] Furthermore, clinical studies involving long-term follow-up of abutment construction using fiber posts have found that the main problem in such treatments is the loss of the post,[12-14] indicating the need to establish a reliable method of fiber-post adhesion.

Ferrari *et al.*[15] bonded silane-treated fiber posts to human teeth using composite resin core and calculated the very small tension of the two surface boundaries. They found that the adhesion strength at the surface boundary between the construction resin and the fiber post was significantly lower than that between the dentin and the construction resin.

Hydrofluoric acid and alumina sandblasting treatments have been proposed as methods for improving the bond strength between fiber posts and composite resin core ; however, the mechanical strength of the fiber posts was impaired with both methods.[16-21] Hence, there is an urgent need to establish a surface treatment method for improving the adhesion strength without mechanically damaging the fiber posts.

It has been reported that low-temperature atmospheric pressure plasma (LT-APP) treatment can be used for surface energy activation and contaminant removal from small gaps without deformation due to physical force or heat.[22] Our previous study reported the effectiveness of pretreatment with LT-APP in improving adhesion.[23] Medard *et al.*[24] reported that plasma treatment improves the adhesion properties of high-polymer materials, such as polyethylene. Thus, LT-APP treatment could similarly improve the adhesion properties of fiber posts.

We investigated the effect of LT-APP treatment of fiber posts on the basis of a three-point bending test and a push-out test. The null hypotheses are : (1) even if the fiber-post surface treatment method differs, there is no difference in the three-point bending strength of the fiber ; (2) even if the fiber-post surface treatment method differs, there is no difference in the adhesion strength between the adhesive resin cement and the fiber post ; and (3) even if the fiber-post surface treatment method differs, there is no difference in the adhesion strength between the composite resin for core build up and the fiber post.

## MATERIALS AND METHODS

### Study design

We investihgated the effect of LT-APP treatment on fiber posts using a three-point bending test, scanning electron microscopy (SEM), and a push-out test. For the three-point bending test, the surfaces of 28 fiber posts 1.4 mm in diameter were treated by different methods. The following four groups of 7 specimens each were established on the basis of the surface treatment conditions : (1) no treatment (control group), (2) alumina blasting (Ab group), (3) LT-APP treatment using He gas (He group), and (4) LT-APP treatment using Ar gas (Ar group). The three-point bending test was then performed to investigate the effect of LT-APP treatment on the three-point bending strength.

SEM examination was done after each surface treatment to investigate the effect of LT-APP treatment on the fiber-post surface structure.

For the push-out test, the surfaces of 84 fiber posts 1.4 mm in diameter were treated by different methods. The following six groups of 7 specimens each were established on the basis of the surface treatment conditions : (1) no treatment (control group), (2) silane treatment only (Pm group), (3) LT-APP treatment using He gas (He group), (4) silane treatment performed after LT-APP treatment using He gas (HePm group), (5) LT-APP treatment using Ar gas (Ar group), and (6) silane treatment performed after LT-APP treatment using Ar gas (ArPm group). After surface treatment, the fiber posts were encapsulated in adhesive resin cement and composite resin core, and then cured. After completion of curing, the centers of the embedded fiber posts were cut in sections 3 mm in thickness for use as the experimental specimens. A push-out test was then performed using the fabricated specimens to investigate the effect of LT-APP treatment on the adhesion strength.

### Materials and devices

The materials and devices used in this study are summarized in Tables 1 and 2, respectively. The specimen fabrication methods for the three-point bending test were as follows. For (1), the specimens were removed from the packaging using tweezers and were not subjected to surface treatment. For (2), alumina particles 50-70 μm in diameter were used to perform alumina blasting for 5 sec at an application pressure of 0.2 MPa and a distance of 30 mm from the target. For (3), pure G2 He gas (Taiyo Nippon Sanso, Tokyo, Japan) was used as the active gas and LT-APP treatment was performed for 30 sec at an application pressure of 0.2 MPa and a distance of 10 mm. For (4), pure G2 Ar gas (Taiyo Nippon Sanso) was used as the ac-

**Table 1** Materials

| Materials | Product name | Manufacturer | Lot No. |
|---|---|---|---|
| Fiber post | Fiber post | GC | 1511171 |
| composite resin core | Unifil Core EM | GC | 1601081 |
| Adhesive resin cement | G-CEM Link Ace | GC | 1611031 |
| Primer | Ceramic Primer Ⅱ | GC | 1503161 |

**Table 2** Devices

| Device | Product name | Manufacturer |
|---|---|---|
| Sandblasting device | Jet Blast Ⅱ | Morita |
| Atomospheric low temperature plasma | A-1000 | Sakigake |
| Universal tester | Autograph AGSJ-5 kN | Shimadzu |
| Scanning electron microscope | S-4800 | Hitachi |
| Ion sputter | E-1030 | Hitachi |

tive gas and LT-APP treatment was performed for 30 sec at 0.2 MPa and a distance of 10 mm.

The three-point bending test was performed using a universal testing machine at a crosshead speed of 1 mm/min. The calculated maximum load was taken to indicate the bending strength. Data processing was performed using material testing operation software (Trapezium version 2 ; Shimadzu, Tokyo, Japan). Statistical analysis was performed using SPSS software version 19 (IBM, Armonk, NY, USA). One-way analysis of variance (ANOVA) was conducted with the surface treatment method as a factor. When a statistically significant difference was found, Bonferroni correction was used to perform multiple comparisons. The level of significance was set at 1%. A field-emission scanning electron microscope (Model S-4800 ; Hitachi, Tokyo, Japan) was used for SEM. After surface treatment under the same conditions as those of the three-point bending test, the fiber posts were coated with platinum using an ion sputter (E-1030 ; Hitachi) to promote conductivity. Ar ion etching was conducted for 30 sec and the platinum was evaporated, thereby yielding the specimens.

The specimen fabrication methods for the push-out test were as follows. For (1), the specimens were removed from the packaging using tweezers and were not subjected to surface treatment. For (2), the specimens were air dried after the addition of each drop of ceramic primer. For (3), pure G2 He gas was used for LT-APP treatment for 30 sec at an application pressure of 0.2 MPa and a distance of 10 mm. For (4), the LT-APP treatment was performed under the same conditions as those in (3), followed by air drying after the addition of each drop of ceramic primer. For (5), pure G2 Ar gas was used for LT-APP treatment for 30 sec at an application pressure of 0.2 MPa and a distance of 10 mm. For (6), the LT-APP treatment was performed under the same conditions as those in (5), followed by air drying after the addition of each drop of ceramic primer. After each surface treatment, the composite resin core or adhesive resin cement was filled in cylindrical frames 8 mm in diameter that were produced using a silicon mold. After insertion of the fiber posts, photopolymerization was done according to the manufacturer's directions. The fiber post was vertically inserted into the silicon mold via a jig manufactured to be parallel to the silicon mold. After curing for 24 hr at room temperature, the centers of the created compound materials were cut into 3-mm-thick slices for use as the experimental specimens (Fig. 1).

A universal testing machine (Autograph AGSJ-5 kN, Shimadzu) was used to perform the push-out test at a crosshead speed of 1 mm/min. The calculated maximum load was taken to indicate the adhesion strength. Statistical analysis of the adhesive strengths obtained with the adhesive resin cement and composite resin core in each surface treatment

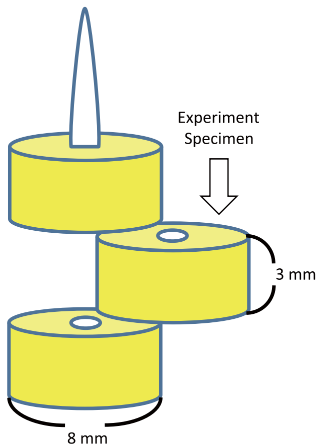


**Fig. 1** Experimental specimen.

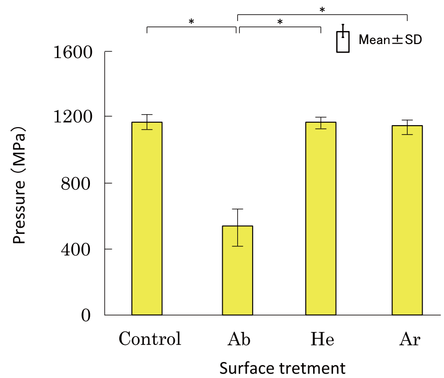


**Fig. 2** Three-point bending strength.
Ab : Alumina blusting,
He : Helium gas treatment,
Ar : Argon gas treatment,
*α<0.01

with each type of gas was performed using SPSS software version 19 (IBM). Two-way ANOVA was conducted with the ceramic primer and LT-APP as factors. When a statistically significant difference was found, Bonferroni correction was used to perform multiple comparisons. The level of significance was set at 1%.

To support the sample size in both the push-out test and the three-point bending test, the effect size was calculated.[25, 26] For the analysis, we used G* Power software version 3.1 (Heinrich Heine University, Dusseldorf, Germany).[27] This study was conducted with Osaka Dental University Research Funds (17-06).

## RESULTS

The results of the maximum bending strength analysis of variance and effective dose based on the three-point bending test are shown in Table 3 and Fig. 2. Statistically significant differences were observed in the surface treatment methods. The maximum bending strength was 1164.9±50.6, 533±113.4, 1152±30.2, and 1134.6±43.7 MPa in the control, Ab, He, and Ar groups, respectively. A representative SEM image is shown in Fig. 3. Although no surface structure damage was observed in the control, He, or Ar groups, surface structure destruction, such as fiber damage, was noted in the Ab group.

The results of the push-out test analysis of variance and effective dose for composite resin core are shown in Tables 4 and 5, and Fig. 4 and 5, respectively. Similarly, the results of the push-out test analysis of variance and the effective dose for adhesive resin cement are shown in Tables 6 and 7, and Figs. 6 and 7, respectively. Statistically significant differences were observed in the ceramic primer and LT-APP for the push-out tests of the composite resin core and for the adhesive resin cement. In the adhesive resin cement, interaction was observed in the cases of both He gas and Ar gas. The bonding strength between the composite resin core and the fiber post was 15.4±0.7, 20.8±0.9, 19.1±1.4, 26.2±3.2, 22.6±1.1, and 27.2±1.5 MPa in the control, Pm, He, HePm, Ar, and ArPm groups, respectively. The bonding strength between

**Table 3** Results of one-way ANOVA of three-point bending test

| Source | Degrees of freedom | Sum of squares | Mean squares | F-value | Effect size ($\eta^2$p) |
|---|---|---|---|---|---|
| Surface treatment | 3 | 2001726.913 | 667242.304 | 125.46 | 0.94 |
| Error | 24 | 127640.324 | 5318.347 | | |
| Total | 27 | 2129367.237 | | | |

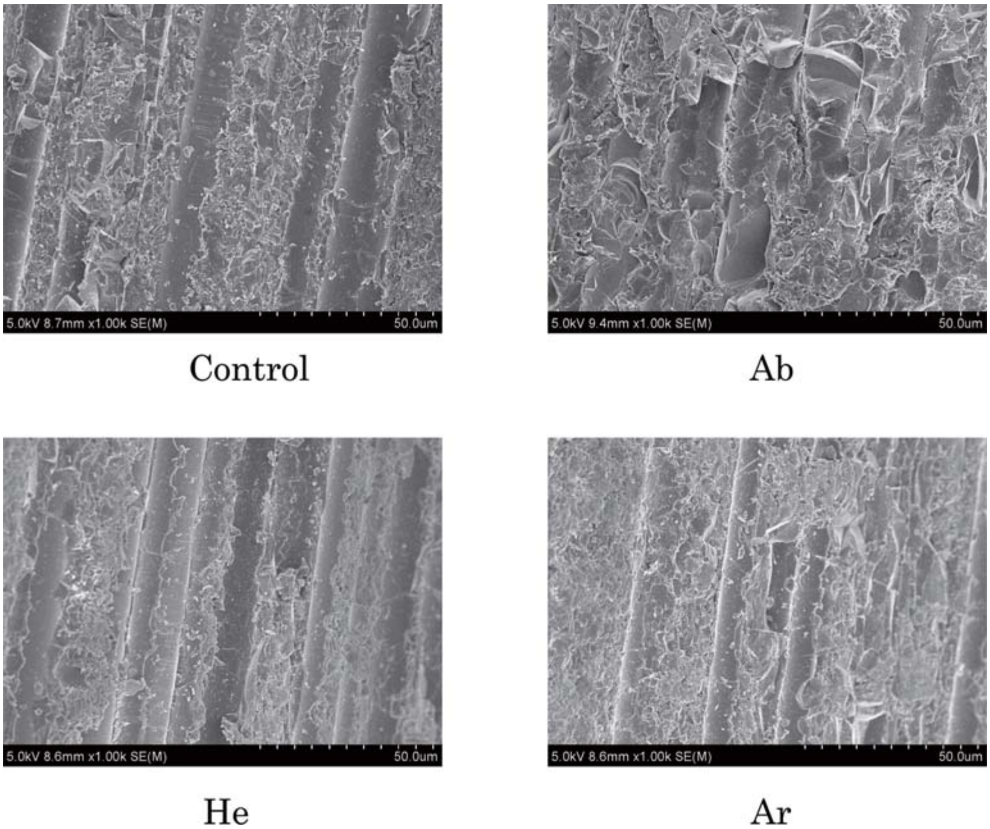


**Fig. 3** Scanning electron microscopic images for the treatments.

**Table 4** Results of two-way ANOVA of bond strength between fiber post and adhesive resin cement for He

| Source | Degrees of freedom | Sum of squares | Mean squares | F-value | Effect size ($\eta^2$p) |
|---|---|---|---|---|---|
| Plasma (He) | 1 | 386.380 | 386.380 | 133.212 | 0.85 |
| Primer | 1 | 331.660 | 331.660 | 114.346 | 0.83 |
| Plasma (He)×Primer | 1 | 32.348 | 32.348 | 11.153 | 0.32 |
| Error | 24 | 69.612 | 2.900 | | |
| Total | 27 | 820.000 | | | |

**Table 5** Results of two-way ANOVA of bond strength between fiber post and adhesive resin cement for Ar

| Source | Degrees of freedom | Sum of squares | Mean squares | F-value | Effect size ($\eta^2$p) |
|---|---|---|---|---|---|
| Plasma (Ar) | 1 | 470.647 | 470.647 | 236.912 | 0.91 |
| Primer | 1 | 300.641 | 300.641 | 151.335 | 0.86 |
| Plasma (Ar)×Primer | 1 | 23.184 | 23.184 | 11.670 | 0.70 |
| Error | 24 | 47.678 | 1.987 | | |
| Total | 27 | 842.150 | | | |

the adhesive resin cement and the fiber post was 17.7±2.1, 22.5±0.8, 23.1±1.6, 32.1±0.5, 24.2±0.5, and 32.5±1.6 MPa in the control, Pm, He, HePm, Ar, and ArPm groups, respectively.

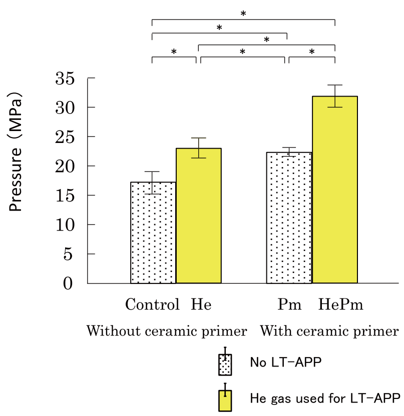


**Fig. 4** Bond strength between fiber post and adhesive resin cement.

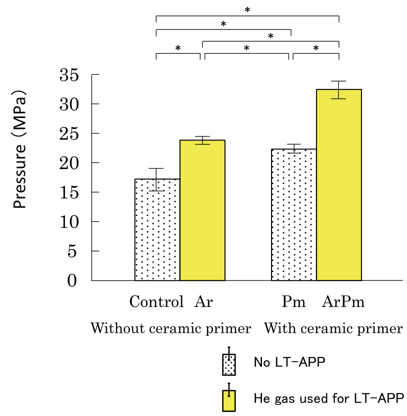


**Fig. 5** Bond strength between fiber post and adhesive resin cement.

**Table 6** Results of two-way ANOVA of bond strength between fiber post and composite resin core for He

| Source | Degrees of freedom | Sum of squares | Mean squares | F-value | Effect size ($\eta^2 p$) |
|---|---|---|---|---|---|
| Plasma (He) | 1 | 30.754 | 30.754 | 14.568 | 0.38 |
| Primer | 1 | 178.751 | 178.751 | 84.670 | 0.78 |
| Plasma (He)×Primer | 1 | 1.155 | 1.155 | 0.547 | 0.02 |
| Error | 24 | 50.668 | 2.111 | | |
| Total | 27 | 261.328 | | | |

**Table 7** Results of two-way ANOVA of bond strength between fiber post and composite resin core for Ar

| Source | Degrees of freedom | Sum of squares | Mean squares | F-value | Effect size ($\eta^2 p$) |
|---|---|---|---|---|---|
| Plasma (Ar) | 1 | 320.852 | 320.852 | 257.258 | 0.91 |
| Primer | 1 | 173.804 | 173.804 | 139.356 | 0.85 |
| Plasma (Ar)×Primer | 1 | 1.590 | 1.590 | 1.275 | 0.05 |
| Error | 24 | 29.933 | 1.247 | | |
| Total | 27 | 526.178 | | | |

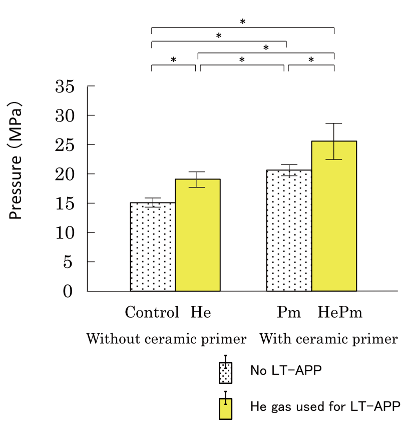


**Fig. 6** Bond strength between fiber post and composite resin core.

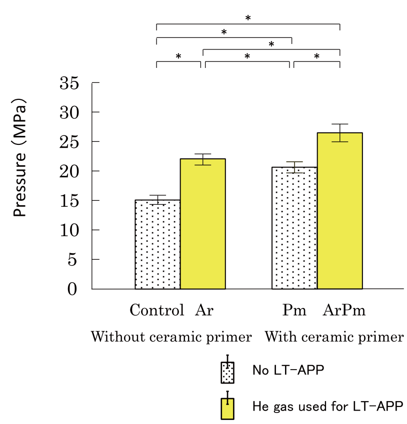


**Fig. 7** Bond strength between fiber post and composite resin core.

## DISCUSSION

The results of the three-point bending testing demonstrated that LT-APP treatment does not alter the three-point bending strength of fiber posts. Therefore, null hypothesis (1) was adopted. Although SEM indicated that alumina blasting clearly caused mechanical damage to the fiber posts, no such damage was caused by LT-APP processing. These results suggest that LT-APP treatment might improve the adhesive strength of fiber posts without causing mechanical damage to them. Therefore, we investigated the bond strength between the fiber post and both the composite resin core and the adhesive resin cement.

Silane treatment is generally applied to the fiber-post surface.[15, 28-30] Fiber posts are composed of fibers consisting of glass and quartz and a matrix bundling these fibers. Through silane coupling treatment, the fibers exposed on the fiber-post surface, adhesive resin cement, and composite resin core are bound via silanol groups. In this experiment, the fiber posts subjected to silane coupling treatment exhibited significantly stronger adhesion strength for both the adhesive resin cement and the composite resin core compared to the untreated specimens, possibly because of effective bonding via silanol groups. The results of this experiment demonstrated that silane coupling treatment after LT-APP treatment improved the adhesion strength between the fiber post and the adhesive resin cement as well as the composite resin core. On the basis of these findings, we were able to reject null hypotheses (2) and (3).

With regard to LT-APP treatment, the active gas undergoes electrolytic dissociation and plasmarization to process the surface at the electron level. Furthermore, it has been reported that the moisture attached to the inside of the gas tube of the active gas used to produce plasma and the moisture in the atmosphere are broken down by high-energy electrons to produce $OH^-$ radicals.[31] It has also been reported that LT-APP treatment severs C-C and C-H bonds, thereby removing contaminants,[32] and it can add the hydrophilic groups C-O and C-OH.[33-35] Thus, LT-APP treatment before silane coupling removed contaminants from the fiber-post surface, improved the fiber-post surface hydrophilicity, and increased the number of exposed fibers. Therefore, the wettability of the ceramic primer on the fiber-post surface also improved, which could have added more silanol groups, resulting in improved adhesion strength.

A comparison of the bonding strengths of the adhesive resin cement and the composite resin core indicated that the adhesion strength of the adhesive resin cement was greater. It has been reported that the factors affecting the viscosity of the resin include filler content, filler shape, filler size, and filler surface treatment conditions.[36-38] The particle diameter of the filler used in the adhesive resin cement was 2-3 μm, whereas that of the filler used in the composite resin core was 5-10 μm. Therefore, it is thought that the adhesive resin cement has lower viscosity than the composite resin core. The improvement in adhesion strength may also be affected by mechanical interlocking force brought about by entry of the adhesive resin cement and composite resin core into the minute concavities between the fibers and the matrix.

The improvement in wettability brought about by LT-APP treatment appeared to indicate higher adhesion strength of the adhesive resin cement because its lower viscosity made it easier to enter the minute concavities between the fibers and the matrix compared to the composite resin core. Through statistical analysis of the adhesive resin cement, interaction was observed when both He gas and Ar gas were used. LT-APP treatment before silane coupling removed contaminants from the fiber-post surface and improved the fiber-post surface hydrophilicity, which could have added more silanol groups. Furthermore, it was assumed that the adhesion resin cement with lower viscosity entered into the micro-recesses between the fibers and the matrix, thereby promoting chemical bonding with the adhesive monomer. The interaction is thought to be achieved through the synergistic effect of the LT-APP and silane coupling treatments.

A comparison of He and Ar indicated stronger adhesion strength when Ar was used as the active gas. As the ionization energy values of He and Ar are 24.5 and 15.8 eV,[31] respectively, He has greater ionization energy than Ar. Thus, ionization of He is more difficult than that of Ar. Plasma treatment is thought to clean the area created by high-energy collisions of the ions with the material surface.[27] More ions were produced when Ar was used as the active gas rather than He. This may have increased the surface treatment efficiency and adhesion strength.

The results suggest that LT-APP treatment is an effective method for processing fiber posts. However, changes over time and the effects on the oral environment after LT-APP treatment have not been investigated. Further studies are warranted for investigating changes over time and simulating the oral environment. In addition, the LT-APP conditions in this experiment were set in accordance with our previous experimental methods ; the optimal conditions for application distance, time, pressure, and active gas type were not investigated. Hence, future studies are warranted to determine the optimal conditions for LT-APP treatment.

## CONCLUSIONS

We investigated the effects of LT-APP treatment on fiber posts using a three-point bending test, SEM, and a push-out test. We concluded that fiber-post properties are not adversely affected by LT-APP treatment, and that it is an effective pre-adhesion treatment for fiber posts.

**Conflict of interest statement**

The authors declare no conflicts of interest.

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