Effects of plasma treatment on the shear bond strength between fiber-reinforced composite posts and resin composite for core build-up

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The aim of this study was to evaluate the effects of plasma treatment on adhesion between fiber-reinforced posts and a composite core material. Two types of posts, methacrylate-based (FRC Postec) and epoxy resin-based (DT Light-Post), were treated with oxygen plasma (O₂), argon plasma (Ar), nitrogen plasma (N₂), or helium mixed with nitrogen plasma (He+N₂) using a radio-frequency generator before bonding to a methacrylate-based composite. Pull-out tests were performed using a universal testing machine. Surface roughness of each group was evaluated using a profilometer. On tensile-shear bond strength, statistical analysis revealed that the type of post, type of plasma treatment, and their interaction significantly influenced the results ($p < 0.05$). Tukey's test revealed significant differences in tensile-shear bond strength between the control and other plasma treatment groups ($p < 0.05$). On surface roughness, Tukey's test revealed significant differences between the control group and the Ar group ($p < 0.05$) with DT Light Post. Plasma treatment appeared to increase the tensile-shear bond strength between post and composite.

Keywords: Fiber-reinforced composite post, Plasma treatment, Tensile-shear bond strength

Received Mar 6, 2009; Accepted Jun 24, 2009

INTRODUCTION

Owing to the growing demand for esthetic restorations in dentistry, tooth-colored, metal-free post and core systems have been developed to overcome the problem of cervical dark shade in endodontically treated teeth, in which metal posts have been used. Recently, quartz and glass fiber-reinforced composite posts (FRCPs) have gained popularity in daily clinical practice and are perceived as promising alternatives to cast posts in overcoming the esthetic problem¹.

The modulus of elasticity of FRCPs is similar to that of dentin² and both can be bonded using an adhesive technique³. Consequently, a homogeneous coronal-radicular reconstruction — so-called “monobloc” condition⁴ — comprising the post, dentin, and composite core can be created. With a “monobloc” condition, stress between the post and dentin generated by occlusal loads can be absorbed by the core and post, and is not transferred to the vulnerable tooth structure⁵.

Although the bonding between the post and root canal dentin plays a pivotal role in the long-term success of a restoration, reliable bonding between the post and the composite core is also indispensable. If the bonding of this interface is poor, debonding and/or fracture of the core and post can occur⁶. Therefore, good adaptation and reliable bonding between the post surface and core build-up material must be achieved⁷.

However, the matrix components of FRCPs are generally epoxy resins or polymers with a high degree

of conversion and highly crosslinked structures, such as urethane dimethacrylate (UDMA) and triethylene glycol dimethacrylate (TEGDMA) monomers⁸. Consequently, there are no functional groups that can react with the methacrylate group in the dental composite resin, resulting in the absence of chemical bonding between these two substrates⁹.

To overcome this problem, several surface treatments of the fiber posts have been undertaken, such as mechanical treatment¹⁰ and chemical treatment^{6,11–13}. These treatments then result in surface microroughness, creating a mechanical interlock between the two surfaces, and/or exposure of the fiber by removal of the matrix, permitting silanization with a silane coupling agent. Some of these treatments may cause detrimental effects when treatment is performed over a long period, such as etching with hydrofluoric acid¹³ or blasting with aluminum oxide particles¹⁰.

Polymer surfaces usually present problems in bonding and finishing due to their low hydrophilicity¹⁴ and low surface energy¹⁵. With plasma treatment, the surfaces of polymers can be improved in terms of hydrophilicity by forming oxygen-containing functional groups, such as C=O¹⁴ and –OH^{16,17}. Plasma is a partially or wholly ionized gas with a roughly equal number of positively and negatively charged particles¹⁸. Plasma consists of energetic species including ions, electrons, free radicals, metastable particles, and photons in the short-wave ultraviolet (UV) range. Surfaces in contact with plasma are bombarded by

these energetic species, such that their energy is transferred from the plasma to the solid surfaces¹⁹. This transferred energy can have various effects on the surfaces of polymers, such as removal of organic contaminants and weak boundary layers by cleaning and ablation^{20–22}; degradation of the polymer chains²³; formation of radicals on the surfaces^{24,25}; alteration of the tacticity of polymer chains²³; creation of a thin crosslinking layer²⁰; and formation of chemical groups on the stabilized surfaces²⁵. These effects result in acid-base interactions and covalent linkages²⁰. Where some of these effects overlap, bonding enhancement is thus successfully achieved through plasma treatment²⁰.

Many studies have revealed improved adhesion of polymers by plasma treatment^{17,26,27}. Plasma surface modification is also appropriate for a variety of biomedical applications¹⁹. In dentistry, only a few plasma applications have been used to improve the adhesive properties of polymers^{28,29}. This is chiefly because it is of utmost importance that the composition of the plasma gas must match the chemical structure of the polymer in order to improve the latter's adhesive properties²⁵. As for plasma treatment on FRCPs, no experimental research has been undertaken to date.

The objective of this study was to evaluate the tensile-shear bond strength between a composite core build-up material and two different types of FRCPs, with and without the four types of plasma treatments. The null hypothesis of the study was that either the post or the plasma treatment has no significant influence on the tensile-shear bond strength between the FRCP and the composite core build-up material.

MATERIALS AND METHODS

Materials used

The materials investigated, including their chemical compositions, manufacturers, and sizes, are listed in Table 1. Two commercial FRCPs were selected: one was methacrylate-based (FRC Postec, Ivoclar Vivadent, Schaan, Liechtenstein; FRC), the other was epoxy

resin-based (DT Light-Post, RTD, St. Egreve, France; DT). These posts were composed of unidirectional quartz fiber (DT) and fiberglass (FRC) embedded in different resin matrices. Both were taper-shaped posts, of which 30% of the coronal portion was parallel.

Plasma treatment groups

FRC and DT posts ($n=45$ each) were randomly divided into five groups ($n=9$ per group): a control group (C), an oxygen plasma treatment group (O_2), an argon plasma treatment group (Ar), a nitrogen plasma treatment group (N_2), and a group treated with a mixture of helium (20%) and nitrogen (80%) plasma ($He+N_2$).

For each plasma treatment group, nine posts were attached to one specimen holder which was placed in the center of the cylindrical quartz chamber of a plasma-generating machine developed by the authors. Under a vacuum with a base pressure of 2.67×10^{-6} MPa, the experimental gas was fed into this chamber at a flow rate of $6 \text{ cm}^3/\text{min}$ and kept at a constant pressure of 1.33×10^{-5} MPa throughout the experiment. The experimental gas was ionized to plasma by inductive coupling discharge, using a radio-frequency generator (Dressler CESAR, Advanced Energy, Stolberg, Germany) at 13.56 MHz, 50 W for 10 minutes.

Specimen preparation for pull-out test

After plasma treatment, each post was kept in a glass vial sealed with a rubber cap at atmospheric pressure and room temperature. Six posts in each group were selected and prepared for a pull-out test within 12 hours of plasma treatment. Each post was placed in an upright position in a special apparatus, consisting of a split metal cylinder with a cylindrical mold in the center of one end. Dimensions of the cylindrical mold were 8 mm in diameter and 2 mm in height (Fig. 1a).

In this upright position, 2 mm of the parallel portion of the coronal end of the tapered post was exposed outside the cylindrical mold. A composite core build-up material (MultiCore Flow, Ivoclar Vivadent,

Table 1 Materials used in the study

Material	Composition	Manufacturer	Lot no.	Size
DT Light-Post	Quartz fiber: 60% vol. Epoxy resin: 40% vol.	Recherches Techniques Dentaires (RTD), St. Egreve, France	093730810	#2, ϕ 1.8 mm, Length=20 mm
FRC Postec	Glass fiber: 61.5% vol. Triethylene glycol dimethacrylate (TEGDMA) and urethane dimethacrylate (UDMA) monomer: 38.5% vol.	Ivoclar Vivadent, Schaan, Liechtenstein	L35052 L47590	#3, ϕ 2 mm Length=20 mm
MultiCore Flow	Base and catalyst of dimethacrylate, inorganic fillers, ytterbium trifluoride, initiator, stabilizer and pigment	Ivoclar Vivadent, Schaan, Liechtenstein	K 34799 K 44769

Fig. 1 Schematic illustration of specimen preparation for pull-out test: (a) fiber-reinforced composite post (FRCP) in the special apparatus; (b) composite resin (CR) on FRCP; (c) metal ring supporting the bottom surface of the composite core; (d) prepared post placed in the center of a new split metal cylinder; (e) the post positioned in the center of the polyvinyl chloride (PVC) tube; (f) the prepared specimen.

Schaan, Liechtenstein) was directly syringed into the mold and around the post to form a core for the pull-out test. Then, the top of the core material was light-activated using a light curing unit (Elipar Trilight, 3M ESPE, Seefeld, Germany) for 40 seconds with a light intensity of 600 mW/cm². After complete polymerization, the post was removed from the mold. Excess core build-up material on top of the specimen was removed with an abrasive paper until the height of this core was exactly 2.0 mm (Fig. 1b). A thin metal ring was placed under the core to support the bottom surface of the core (Fig. 1c).

The prepared post was then placed upside down in the center of a new split metal cylinder with a cylindrical mold in the center of the upper half. Dimensions of the cylindrical mold were 18 mm in diameter and 5 mm in height, so that the apical 8 mm of the post was in the center of this mold (Fig. 1d). A polyvinyl chloride (PVC) tube with a diameter of 18 mm and a height of 10 mm was inserted into this mold (Fig. 1e). An autopolymerized resin (Instant Tray Mix, Lang Dental Manufacturing Co., Wheeling, IL, USA) was mixed and poured into the tube until it reached the top of the tube. In this way, the post in the tube was embedded in the autopolymerized resin. After complete polymerization, the specimen was detached from the mold (Fig. 1f) and kept in a glass vial sealed with a rubber cap at atmospheric pressure and room

temperature for 24 hours before the pull-out test.

For the pull-out test, the PVC tube was attached to a special jig holder of a universal testing machine (LR10K, Lloyd Instruments, Fareham, UK). The thin metal ring under the composite core was supported by a special upper jig holder, fixed in the tensile device of the machine's moving part. Each specimen was loaded in tension at a crosshead speed of 1mm/min until the composite resin core was separated from the post. The maximum load (N) was divided by the bonding area (mm²) and then recorded in MPa for statistical analysis. After the pull-out test, all debonded interfaces between the post and the composite core build-up material were observed using a light microscope (Meiji Techno Co. Ltd., Tokyo, Japan) at $\times 15$ magnification.

Surface roughness measurement

The remaining three posts in each group were evaluated for surface roughness by a profilometer (Tyloscan 150, Taylor Hobson, Leicester, UK), using a scan field of 2 $\times$ 0.5 mm on three randomly selected areas of each post. The average surface roughness (S_a) of the posts in each group was expressed in μ m.

Statistical analysis

Statistical analyses of tensile-shear bond strength and surface roughness were performed respectively using two-way ANOVA. Tukey's test was used for *post hoc*

multiple comparisons to detect significantly different pairs. In all the tests, the level of significance was set at $p < 0.05$, and calculations were performed using a statistical software (SPSS ver 13.0, SPSS Inc., Chicago, IL, USA).

RESULTS

Tensile-shear bond strength

The tensile-shear bond strength values (MPa), obtained in the present study, are displayed in Fig. 2. Two-way ANOVA revealed that the tensile-shear bond strength between the post and composite core build-up material was significantly influenced ($p < 0.01$) by the type of post, type of plasma treatment, and their interaction. Among the treatment groups, Tukey's test revealed significant differences between the control group and the other plasma treatment groups. Besides, a significant difference in tensile-shear bond strength was detected between the O₂ and the other plasma

Fig. 2 Tensile-shear bond strengths (MPa) for all groups displayed in a box and whisker plot.

Table 2 Means (μm) $\pm$ s.d. of surface roughness calculated for all the treatment groups

Type of post	Treatment				
	Control	O ₂	Ar	N ₂	He+N ₂
FRC	0.83 $\pm$ 0.06 ^a	1.04 $\pm$ 0.10 ^a	1.23 $\pm$ 0.16 ^a	1.17 $\pm$ 0.12 ^a	0.96 $\pm$ 0.06 ^a
DT	0.73 $\pm$ 0.30 ^b	0.70 $\pm$ 0.26 ^b	1.14 $\pm$ 0.15 ^c	0.93 $\pm$ 0.15 ^{b,c}	0.85 $\pm$ 0.32 ^{b,c}

Values with different superscript letters denote statistically significant differences at $p < 0.05$.

Fig. 3 Light microscopic images of the debonded surfaces of FRC and DT posts: (a, b) debonded surfaces of FRC Postec; (c, d) debonded surfaces of DT Light-Post.

treatment groups. For the FRC posts, one-way ANOVA revealed that all of the plasma treatments significantly influenced the tensile-shear bond strength when compared with the control group ($p < 0.05$). For the DT posts, only the Ar, N₂, and He+N₂ treatments significantly influenced the tensile-shear bond strength when compared with the control group ($p < 0.05$).

Surface roughness

Table 2 shows the surface roughness results of the FRC and DT posts in all the treatment groups. Two-way ANOVA revealed that the average surface roughness on the surface of the post was significantly influenced by the type of post and type of plasma treatment, but not by their interaction. Among all the treatment groups, Tukey's test revealed significant differences in surface roughness between the control and Ar treatment groups and between the O₂ and Ar treatment groups. For the FRC posts, one-way ANOVA revealed no significant differences in surface roughness between any of the groups. However, for the DT posts, there was a significant difference between the O₂ and Ar treatment groups.

Light microscopic analysis

Light microscopic images of the debonded specimens after the pull-out test demonstrated that mixed adhesive/cohesive failure at the composite core-build-up material/post occurred in both types of posts (Fig. 3).

DISCUSSION

In the present study, for both FRC and DT posts, treatment with Ar, N₂, and He+N₂ plasma significantly improved the tensile-shear bond strength between the post and composite core build-up material when compared with the control group ($p < 0.05$). However, the O₂ treatment significantly improved the tensile-shear bond strength only for the FRC posts. Generally, with O₂ treatment, polar functional groups can be induced on the surface of the polymer, resulting in higher surface wettability^{20,22}. Therefore, results of this study suggested that the induction of polar functional groups probably did not apply to the O₂-treated DT posts. Bond breaking or degradation may be difficult to achieve by O₂ treatment in the highly crosslinked networks of DT posts, which were composed of epoxy resin. On surface roughness, oxygen plasma treatment usually creates microroughness on the treated surface due to an etching effect^{21,22}. However, in the present study, no significant increases in average surface roughness were observed on the surface of either the FRC or DT post when compared with the control groups. This was probably due to the low concentration of O₂ in this study.

Argon plasma treatment significantly enhanced the tensile-shear bond strength for both types of posts. This enhancement might be the result of polymer chain scission caused by the bombardment of energetic Ar particles, which were high-molecular-weight particles.

Although inert gas plasma treatments (He, Ar) do not induce any new reactive functionalities on the polymer surface²⁵, treatment with inert gas can induce free radical formation on the polymer surface through ion bombardment and ultraviolet (UV) radiation²⁴. These free radicals can react with other surface radicals or with other chains in the chain-transfer reactions of polymers.

By these reactions, chemical interaction between free radicals on the surface of the FRCPs and the functional groups in the composite core build-up material may occur, thereby resulting in a significant increase in tensile-shear bond strength. It could be assumed that these chemical interactions, rather than micromechanical interlocking between the two surfaces, were the cause of a higher tensile-shear bond strength in the FRC posts, because the average surface roughness of the FRC posts was not increased.

In N₂-treated groups, the tensile-shear bond strengths between the composite core build-up material and both types of posts were significantly increased. The increase in bond strength might be the result of functional groups induced on the surfaces of both types of posts. Grace and Gerenser²⁵ demonstrated that the amine and imine carbon species were induced as functional groups on the surface of N₂-treated polystyrene. The majority of these functional groups usually contain a terminal nitrogen (primary amine or imine). Nitrogen plasma treatment also damaged the polymer surface through bond breaking of the phenyl ring in polystyrene. In the present study, it was hypothesized that the induced functional groups on the surfaces of both N₂-treated FRC and DT posts might contain a terminal nitrogen, which then reacted with the functional groups in the composite core build-up material. Moreover, these amine and imine functional groups might remain stable over time as compared to the oxygen functional groups, this speculation is well confirmed by a study on the aging effects of N₂-modified PET²⁵.

Amongst all the plasma treatment groups, the He+N₂ treatment yielded the highest tensile-shear bond strength between the composite core build-up material and both types of posts, but it was not significantly different from that in the Ar group. Liston *et al.*²⁰ reported that the helium plasma in this mixture strongly emitted vacuum ultraviolet (VUV) radiation, which could have introduced a subsurface crosslinking layer. This layer then acted as a barrier against diffusion of low-molecular-weight species from the polymer bulk to the surface. On the other hand, this layer could have reduced the thermodynamically driven re-orientation of polar moieties away from the surface into the subsurface²⁵, which meant that surface energy might still have remained. Moreover, as a result of surface bombardment by the energetic species of helium plasma, selective bond breaking could have occurred. Subsequently, with N₂ treatment in this study, the nitrogen functional groups could have been easily induced on the surface and become more stable

over time. These findings were in agreement with those of Grace and Gerenser²⁵⁾, who pointed out that the degree of incorporation of new functional groups with reactive/inert gas mixtures depended on the efficiency of the VUV radiation emitted by the gas.

With the FRC posts, the active sites on the polymer surface and the base monomer of the composite core build-up material were the same. Therefore, chemical interaction between the functional groups and/or free radicals in both materials occurred and resulted in high tensile-shear bond strength. This was reflected in the mixed adhesive/cohesive failure mode, resulting in exposure and delamination of fiber from the post. For the DT posts, the polymer matrix was epoxy resin which was completely different from the composite core build-up material. The epoxy resin is a highly crosslinked material, such that chemical interaction between the plasma-treated post and the composite core build-up material was difficult to achieve. It is interesting to observe that the mixed failure mode exhibited by this group demonstrated low tensile shear-bond strength. This might stem from the poor bond between the quartz fiber and the epoxy resin or surface topography of the post. At this juncture, it must be clarified that many important factors come into play in each plasma treatment when it is applied to optimize the adhesive properties of polymers. This present study evaluated only two types of fiber posts in dry condition. Hence, further studies are needed on the parameters of plasma treatment, hydrothermal stability of the interface, and the aging effects of plasma-treated posts.

Based on the results obtained in this study, the null hypothesis that either the post or plasma treatment has no significant influence on the tensile-shear bond strength between the FRCP and the composite core build-up material was rejected.

CONCLUSIONS

Within the limits of the present study, it was concluded that both the type of post and the type of plasma treatment significantly influenced the tensile-shear bond strength between the composite core build-up material and plasma-treated FRCPs ($p < 0.05$). For the FRC posts, O₂, Ar, N₂, and He+N₂ treatments significantly improved the tensile-shear bond strength. For the DT posts, the Ar, N₂, and He+N₂ treatments significantly improved the tensile-shear bond strength ($p < 0.05$). On surface roughness, there were significant differences between the control group and the Ar group and between the O₂ and Ar groups ($p < 0.05$) for the DT posts.

ACKNOWLEDGMENTS

The authors would like to sincerely thank Mr. Chanchai Umongno of the Fast Neutron Research Facility Center, Physics Department, Faculty of Science, Chiang Mai University, Thailand, for his

technical assistance and advice, Dr. Narumanas Korwanich, Faculty of Dentistry, Chiang Mai University, Thailand, for his statistical advice, and Dr. M. Kevin O Carroll, Professor Emeritus of the University of Mississippi, School of Dentistry, USA and Faculty Consultant at Chiang Mai University, Faculty of Dentistry, Thailand, for his English editorial assistance in the preparation of the manuscript.

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