The adhesion between fibre posts and composite resin cores: the evaluation of microtensile bond strength following various surface chemical treatments to posts

M. Vano[1,2], C. Goracci[1], F. Monticelli[1], F. Tognini[1], M. Gabriele[2], F. R. Tay[3] & M. Ferrari[1]

[1]Department of Restorative Dentistry and Dental Materials, University of Siena, Siena, Italy; [2]Department of Oral Surgery, University of Pisa, Pisa, Italy; and [3]Faculty of Dentistry, University of Hong Kong, Pokfulam, Hong Kong SAR, China

Abstract

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Aim To evaluate the influence of various surface treatments to fibre posts on the microtensile bond strength with different composite resins.

Methodology A total of 110 fibre posts were randomly divided into five groups, according to the surface pre-treatment performed. Group 1: immersion in 24% H_2O_2 for 10 min and silanization for 60 s; group 2: immersion in 10% H_2O_2 for 20 min and silanization for 60 s; group 3: immersion in 4% hydrofluoric acid gel for 60 s and silanization for 60 s; group 4: silanization of the post surface for 60 s and application of the bonding agent G-Bond; group 5: silanization of the post surface for 60 s (control group). After treatment, two posts were randomly selected from each group to evaluate the morphological aspect of the post surface with scanning electron microscopy. The remaining posts in each group were divided into five subgroups of five posts each, which differed in the type of composite resin used for the core build-up. Post-core strength were calculated and the differences among experimental groups were analysed with two-way ANOVA and the Tukey test ($\alpha = 0.05$).

Results The post-core strengths achieved in groups 1 and 2 were significantly higher ($P < 0.05$), than those of groups 3, 4 and 5. The post-core strength in the control group was significantly lower ($P < 0.05$) than all other groups.

Conclusions Hydrogen peroxide and hydrofluoric acid both modified the surface morphology of fibre posts and with silane, significantly enhanced the interfacial strength between them and core materials.

Keywords: fibre posts, hydrofluoric acid gel, hydrogen peroxide, microtensile bond strength, surface treatment.

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Introduction

The restoration of root filled teeth often requires the placement of a post to ensure adequate retention of the core (Gutmann 1992). First introduced in 1990 (Duret *et al.* 1990), fibre posts were rapidly accepted by clinicians (Ferrari *et al.* 2000b), and provided a viable alternative to cast metal posts for the restoration of root filled teeth. The major advantage of fibre posts is their similar elastic modulus to dentine, producing a stress field similar to that of natural teeth, whereas metal posts exhibit high stress concentrations at the post-dentine interface (Pegoretti *et al.* 2002). Clinical studies have demonstrated high success rates without the occurrence of root fractures (Ferrari *et al.* 2000a,b). Moreover fibre posts are ready to use whereas the

Correspondence: Marco Ferrari, Chairman and Professor, Department of Restorative Dentistry and Dental Materials, University of Siena, Policlinico Le Scotte, Viale Bracci, 53100 Siena, Italy (Tel.: +393384623264; fax: +390577233117; e-mail: ferrarimar@unisi.it).

construction of a cast post and core is more time consuming and demands extra clinic and laboratory time (DeSort 1983).

In vivo data have shown that the establishment of reliable bonds at the root-post-core interfaces are critical for the clinical success of a post-retained restoration (Monticelli *et al.* 2003). It has also been demonstrated that parameters such as post length, shape, and post-surface characteristics influence post retention (Schwartz & Robbins 2004).

In order to improve the bond strength between the post and the resin cement, many surface pre-treatment procedures for posts have been investigated that involve the use of mechanical (Kern & Thompson 1994, Sahafi *et al.* 2003) or chemical agents (Kern & Wegner 1998, Yangida *et al.* 2001). Chemical treatment is aimed at roughening the post surface, thus enhancing the mechanical interlocking between post and resin cement (Wolf *et al.* 1993). In a recent *in vitro* study, post surface pre-treatment with hydrogen peroxide has been shown to significantly increase the bond strength between fibre posts and flowable materials used for core build-up (Monticelli *et al.* 2005b).

Hydrofluoric acid in combination with a silane-coupling agent is often employed to enhance the bond strength between composite resins and feldspathic ceramics (Hayakawa *et al.* 1992, Aida *et al.* 1995, Chen *et al.* 1998, Ozcan & Vallitu 2003). Silanes are also used for coupling the glass filler particles or the glass fibres with the embedding matrix in composite and fibre-reinforced resins respectively (Ishida 1985, Iglesias *et al.* 2002). Silane coupling agents are able to chemically bridge resins and OH-covered inorganic substrates. At the fibre post-composite core interface, chemical coupling is only possible between the resin of the core material and the exposed glass fibres of the post (Ferrari & Scotti 2002, Aksornmuang *et al.* 2004, Goracci *et al.* 2005). Because of the difference in chemistry, no bonding is expected to occur between the methacrylate-based resin of the core and the epoxy resin of the fibre post matrix (Monticelli *et al.* 2005b).

Several materials have been used for core build-ups that differ in their mechanical properties, viscosities and setting reactions (Combe *et al.* 1999). In a recent microscopic study (Monticelli *et al.* 2005a), flowable composites achieved structural homogeneity and continuity with the post surface that were superior to hybrid composites. However, the latter materials are expected to provide higher mechanical properties than the lightly filled flowable composites. Also, several composite resins specifically formulated for abutment build-up are currently available in the market.

Previous studies (Goracci *et al.* 2005, Monticelli *et al.* 2005b) have shown that hydrogen peroxide is able to dissolve the epoxy resin matrix, breaking epoxy resin bonds and exposing the surface of fibres to silanization. This method of pre-treatment was found to be effective for enhancing the retention between epoxy resin-based, conventional fibre post systems and core materials (Monticelli *et al.* 2005b). However, little is known of the physical and chemical effects of hydrogen peroxide on methacrylate-based resin fibre post systems.

The present study was aimed at evaluating the influence of post-surface treatment with hydrofluoric acid or hydrogen peroxide on the microtensile bond strength between glass fibre posts containing methacrylate resin and different composite resins for core build-up. The changes in post-surface characteristics following the different pre-treatments were also observed using scanning electron microscopy (SEM). The tested null hypotheses were: (i) the microscopic aspect of the post surface and the post-core strength are not affected by different post surface pre-treatments; (ii) the type of resin composite used for core build-up has no influence on the post-core interfacial strength.

Materials and methods

One hundred and ten translucent glass fibre posts (GC Corporation, Tokyo, Japan) with a maximum diameter of 1.6 mm were used in the study. They are made of unidirectional glass fibres (77% vol) bound in a methacrylate resin matrix (23% vol). Posts were randomly picked from the boxes and divided into five groups of 22 each, depending on the post surface pre-treatment to be performed. These pre-treatments include: immersion in 24% hydrogen peroxide for 10 min at room temperature and silanization for 60 s (group 1); immersion in 10% hydrogen peroxide for 20 min at room temperature and silanization for 60 s (group 2); immersion in 4% hydrofluoric acid gel (Porcelain Etchant, Bisco, Schaumburg, IL, USA) for 60 s and silanization for 60 s (group 3); silanization of the post surface for 60 s and application of the bonding agent G-Bond (GC Corp.) (group 4); silanization of the post surface for 60 s only (group 5, control group). After the application of hydrogen peroxide or hydrofluoric acid, all the posts were rinsed with water and air-dried. The silane-coupling agent (Monobond-S;

Ivoclar-Vivadent, Schaan, Liechtenstein) was applied in a single layer with a brush on the post surface, and left to air dry for 60 s at room temperature. The chemical composition and batch numbers of the tested materials are reported in Table 1.

SEM analysis

Two posts were randomly selected from each group for SEM examination of the superficial aspect of the post following surface pre-treatment. In each group, one post was observed longitudinally, while the other one was cross-sectioned by means of a water-cooled diamond blade (Isomet 1000; Buehler, Lake Bluff, IL, USA). All the posts were sonicated for 5 min in deionized water (CP104; CEIA Int., Rassy CDG, France), immersed in 96% ethanol, and gently air dried. Each post was mounted on a metallic stub, gold-sputtered (Polaron Range SC7620; Quorum Technology, Newhaven, UK), and observed under a JSM 6060 LV microscope (JEOL, Tokyo, Japan) at different magnifications (200×, 1000×).

Core build-up and microtensile test procedures

The materials used for core build-up were: two flowable composites UniFil Flow (subgroup A) and UniFil Lo Flo Plus (subgroup B), the hybrid composite Gradia Direct (subgroup C), and the core material UniFil Core (subgroup D). These materials were handled according to the instructions supplied by the manufacturer (GC Corp.).

For the core build-up procedure, each post was positioned upright on a glass slab, and secured with a drop of sticky wax. A cylindrical plastic matrix was then placed around the post and adjusted so that the post would be exactly in the middle. The matrix was 10 mm in diameter and the length was equal to the nontapered portion of the post. For an easier calculation of the bonding surface in microtensile specimens, it is desirable that the post diameter be constant throughout the post length (Goracci *et al.* 2005).

The light-activated composites were applied to the post in 1 to 2-mm thick increments. Each increment was carefully placed onto the post surface, and light-

Table 1 List of investigated materials

Material	Batch number	Composition	Manufacturer
Post			
GC fibre post	100602061	Glass fibres (77% vol), methacrylate resin matrix (23% vol)	GC Corporation, Tokyo, Japan
Core material			
UniFil Flow	0309101	Di-2-methacryloyloxyethyl, 2,2,4-trimethylhexamethylene dicarbamate, triethylene glycol dimethacrylate, fluoro-alumino silicate glass (50–60%), silica powder 10–15%	GC Corporation, Tokyo, Japan
UniFil Lo Flo Plus	0405131	Urethane dimethacrylate, triethylene glycol dimethacrylate, fluoro-alumino silicate glass (30–40%), silica powder 5–10%, camphorquinone	GC Corporation, Tokyo, Japan
Gradia Direct	0305151	Urethane dimethacrylate, dimethacrylate comonomers, silica, pre-polymerized filler, pigments, catalysts	GC Corporation, Tokyo, Japan
UniFil Core	0310162	Urethane dimethacrylate, dimethacrylate, photo/chemical initiator, fluoro-amino silicate glass	GC Corporation, Tokyo, Japan
Surface treatment			
Monobond S	E26882	1% wt 3-methacryloxypropyltrimethoxysilane (3-MPS), ethanol/water-based solvent	Ivoclar-Vivadent, Schaan, Liechtenstein
Porcelain Etchant	0300012353	4% Hydrofluoric acid gel	Bisco, Schaumburg, IL, USA
Hydrogen peroxide 24%	073196	24% Hydrogen peroxide	Sella, Schio, Italy
Hydrogen peroxide 10%	12	10% Hydrogen peroxide	Nova Argentia, Milano, Italy
G-Bond	0411221	4-methacryloyl-oxyethyl trimelliate monomer, phosphoric acid ester monomer	GC Corporation, Tokyo, Japan

cured separately for 40 s according to the manufacturer's instructions, using a halogen light curing unit with an output of 600 mW cm^{-2} (VIP; Bisco, Schaumburg, IL, USA). The composite was always irradiated directly from the open upper side of the matrix and through the post. Irradiation was never performed through the plastic matrix. Once the matrix was completely filled, the composite cylinder was detached from the glass slab. An additional 40 s irradiation was then performed from the bottom of the cylinder prior to the removal of the matrix, to ensure optimal polymerization of the core material.

The sectioning and loading of the specimens began on completion of the core build-up procedure, in order to simulate the clinical situation of immediate loading following core build-ups. Each composite cylinder was secured on an Isomet cutting machine for sectioning (Buehler). Two longitudinal cuts were initially made with the water cooled diamond blade along the two opposite sides of the post at its outermost periphery. This sectioning produced a rectangular slab of uniform thickness, with the post in the centre and the core build-up composite on either side. Each slab was subsequently sectioned into 1-mm thick sticks for microtensile bond testing (Fig. 1).

Each stick was secured with cyanoacrylate adhesive (Zapit, Dental Ventures of America, CA, USA) to the two free sliding components of a jig, which was mounted on a universal testing machine (Controls, Milan, Italy). The stick was loaded in tension at a cross-head speed of 0.5 mm min^{-1} until failure occurred at either side of the post-composite interface (Reis *et al.* 2004). Bond strength was expressed in MegaPascals (MPa), by dividing the load at failure by the bonding surface area. As the bonded interface was curved, its area was calculated using a mathematical formula previously applied by Bouillaguet *et al.* (2003).

Statistical analysis of the microtensile bond strength data

After analysing the bond strength data for the normality of data distribution (Kolmogorov–Smirnov test) and homogeneity in variances (Levene's test), a two-way ANOVA was applied with bond strength as the dependent variable, and the types of surface pre-treatment and core material as factors. The Tukey test was used for *post hoc* multiple comparisons of surface pre-treatments and core materials. In all the tests, the level of significance was set at $\alpha = 0.05$, and calculations were handled by the SPSS 11.0 software (SPSS Inc.; Chicago, IL, USA).

Results

Microtensile bond strength test

The means and standard deviations of the bond strengths for the five experimental and control groups are shown in Table 2. Statistical analysis revealed that both the post-surface treatment procedure and the type of composite resin used for core build-up had significant influence on microtensile bond strength ($P < 0.05$). More precisely, the post-core strengths achieved following the two variants of hydrogen peroxide pre-treatment (groups 1 and 2) were comparable and significantly higher than those of groups 3, 4 and 5 in which the post surface had been treated with 4% hydrofluoric acid/silane, silane/bonding agent and silane (control group) respectively. In the control group (group 5), the lowest post-core strength was achieved, and the difference was statistically significant ($P < 0.05$).

In groups 1 and 2, the post-core bond strengths were similar regardless of the composite resin used for the

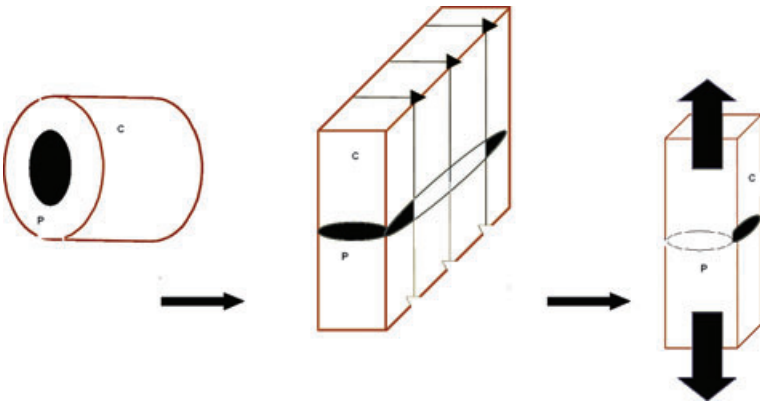

Figure 1 A schematic of the sectioning procedure. One-millimetre thick sticks were serially cut from the slab (C = core, P = post).

Table 2 Mean and standard deviation (in parenthesis) of post-core bond strength calculated for all the experimental groups

	Bond strengths in MPa (SD) after surface pre-treatments				
Core material	Silane for 60 s	Silane for 60 s + G-Bond	24% H_2O_2 for 10 min + Silane for 60 s	10% H_2O_2 for 20 min + Silane for 60 s	4% Hydrofluoric acid gel for 60 s + Silane for 60 s
UniFil Flow	5.02 (0.95)	6.04 (2.06)	13.75 (3.20)	13.44 (2.26)	8.55 (3.26)
UniFil Lo Flo Plus	5.88 (1.13)	6.37 (2.01)	14.93 (3.03)	13.82 (3.32)	9.66 (2.94)
Gradia Direct	7.07 (1.2)	7.48 (2.41)	14.54 (3.36)	13.62 (3.38)	10.96 (3.21)
UniFil Core	8.29 (1.79)	8.53 (2.95)	15.35 (3.37)	14.49 (3.22)	12.78 (2.63)

core build-up (Table 2). Conversely, core material was a significant factor in groups 3, 4 and 5 with UniFil Core recording the highest bond strengths ($P < 0.05$). In addition the difference between Gradia and UniFil Flow was significant ($P < 0.05$) in groups 4 and 5 (Table 2).

SEM analysis

SEM evaluation revealed that the post surface morphology was modified following treatment with hydrogen peroxide and hydrofluoric acid. The two variants of treatment with hydrogen peroxide produced similar changes in the ultrastructure of the post surface. At a lower magnification (Fig. 2a and 3a), a uniform distribution of micro-spaces was evident among the exposed fibres. As a result, a rough surface along the whole post length was created. Exposed fibres did not appear to be damaged by the action of hydrogen peroxide and no defects or fractures were evident on their surfaces (Fig. 2b and 3b). The cross-sections revealed a significant exposure of the superficial fibres because of resin matrix removal, especially following 24% hydrogen peroxide treatment for 10 min (Fig. 2c and 3c). However, the resin matrix was retained in the spaces among the inner fibres.

Treatment with 4% hydrofluoric acid had a greater impact on the post structure. The resin matrix was removed more extensively and to a greater depth (Fig. 4a). Some fibres appeared to be thinner (Fig. 4a,c), and damaged (Fig. 4b). Cross-sections of the posts revealed that the outermost glass fibres were deprived of their resin embedding to a greater extent (Fig. 4c).

Discussion

The bond strengths of different composite resins to translucent glass fibre posts were affected by both the core material and by the type of post surface pre-treatment. Moreover, SEM revealed that the post pre-treatments under investigation had an impact on post surface characteristics. Thus, the null hypotheses tested in this study can be rejected.

Hydrogen peroxide was found to be the most effective treatment with respect to post-core bond strength. In fact, either concentration of hydrogen peroxide significantly enhanced the interfacial bond strength between fibre posts and core materials ($P < 0.05$). These data are in agreement with the results of previous microtensile tests by Monticelli *et al.* (2005b). In particular, post-core bond strengths in group 1 and 2 were very similar, regardless of the material used for core build-up (Table 2).

Interestingly, the flowable composite groups benefited the most from post surface pre-treatment with hydrogen peroxide. It can be speculated that because of their low viscosity, the flowable composites were able to penetrate optimally within the post surface irregularities, taking the greatest advantage of the increase in surface area available for bonding following post surface pre-treatment. This enabled the flowable composites to achieve a bond with the post that was as solid as that established by intrinsically stronger composites, such as Gradia Direct and UniFil Core.

The depths of resin removed from the matrices of the fibre posts were similar for the two concentrations of hydrogen peroxide (Fig. 2c and 3c). Post-core bond strengths were also increased as a result of post treatment with 4% hydrofluoric acid, though to a lesser extent than following post immersion in hydrogen peroxide. One conceivable explanation for these results could be that hydrofluoric acid selectively dissolves the glass component of the fibre post, producing an irregular pattern of microspaces on the post surface (Fig. 4a,b). This may increase the surface area and facilitate the penetration of the composite, especially the flowable resins, into the microretention of the etched post surface. Hydrofluoric acid etching has been found to improve the bond strength between resin and conventional silicate-based ceramics (Stangel *et al.* 1987, Wolf *et al.* 1993). However, this study, in

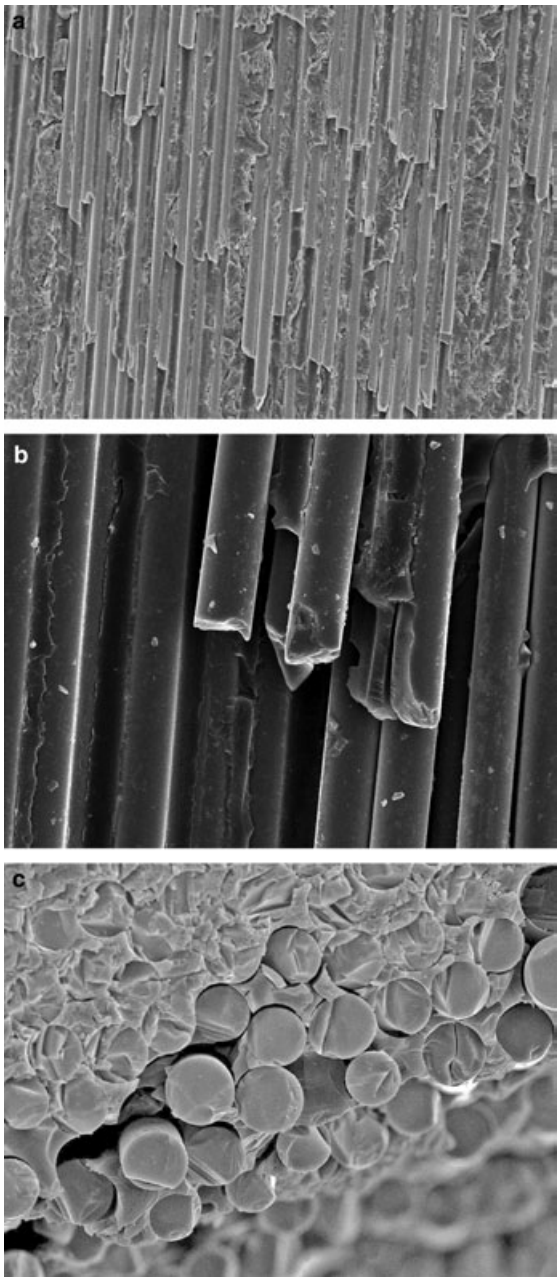

Figure 2 SEM images of the post surface after treatment with 24% hydrogen peroxide for 10 min (a) (200× bar = 100 μm), (b) (1000×, bar = 10 μm). Cross section of the post (c) (1000×, bar = 10 μm).

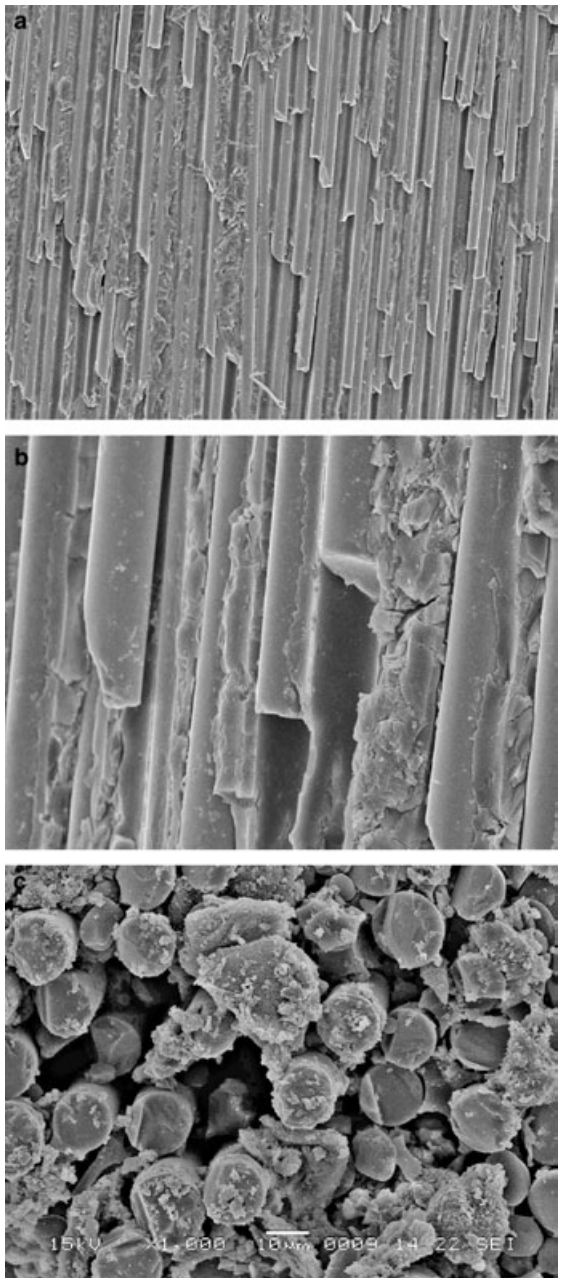

Figure 3 Representative SEM micrographs of the post surface treated with 10% hydrogen peroxide for 20 min: (a) (200×, bar = 100 μm), (b) (1000×, bar = 10 μm). Cross-section of the post (c) (1000×, bar = 10 μm).

agreement with a previous report (Dallari & Mason 2004), showed that hydrofluoric acid alters the post structure more radically. Conversely, for hydrogen peroxide pre-treatment, SEM analysis revealed a differential removal of the resin matrix instead of the glass fibre component, leaving denuded, intact fibres that appeared undamaged.

This study also evaluated the use of a single-component silane coupling agent with and without a bonding agent. The results clearly showed that in the

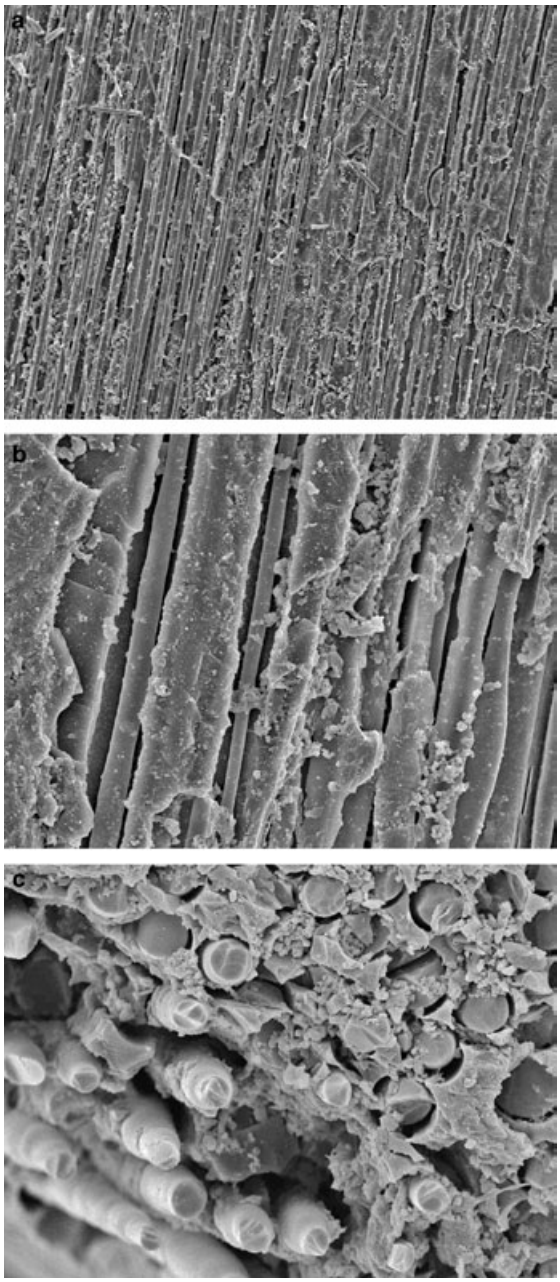

Figure 4 SEM images of the post surface after treatment with 4% hydrofluoric acid gel for 60 s: (a) (200× bar = 100 μm), (b) (1000×, bar = 10 μm). Cross section of the post (c) (1000×, bar = 10 μm).

absence of surface modification of the post surface, the adjunctive use of an adhesive only produced limited improvement in the coupling of resin composites to even methacrylate resin-based fibre posts. Silane coupling agents mainly exert their function by bonding chemically to the posts and core material and improving surface wettability (Plueddemann 1991). Following the manufacturer's specifications, the silane was applied in a single layer. According to the results of a recent *in vitro* study, the formation of a multilayer structure may result in a reduction of the effectiveness of silane coupling, as the number of free methacrylate groups is reduced, and cohesive failure within the silane coating may occur (Debnath *et al.* 2003). The low bond strength values obtained for group 4 and 5 may be due to the absence of free radicals in the pre-polymerized post that is performed under heat and vacuum by the manufacturer. As an oxygen inhibition layer is absent, the bonding is poor.

The method utilized for bond strength testing was the microtensile bond test that has been reported to be suitable for the evaluation of interfacial bond strengths on areas below 1 mm^2 (Pashley *et al.* 1999). In particular, the nontrimming variant of the technique was adopted to reduce the number of premature failures during specimen preparation, in comparison with the 'more aggressive' trimming variant of the microtensile bond test (Goracci *et al.* 2004b).

However this experimental technique has some limitations: The data of this *in vitro* study does not give an exact prediction whether the *in vitro* performance of the fibre posts is the same as the performance *in vivo*. Only one type of fibre post was tested in this study. It would be of interest to analyse other types of posts and to compare their performances.

In this *in vitro* study the pre-treatment of the post was immediately followed by the application of the resin composite for the core build-up. Further *in vitro* and *in vivo* studies are necessary to evaluate whether the positive effect on post-core bond strength is still retained by pre-treating the post surface well in advance of the clinical use. Evaluation of such a strategy will enable manufacturers to supply pre-treated fibre posts in pre-sealed sachets, as well as saving clinicians valuable chair-time.

Conclusions

Surface treatment of posts with hydrogen peroxide and silane application or hydrofluoric acid and silane application significantly enhances the interfacial bond strength between fibre posts and core materials. Post pre-treatment with 24% hydrogen peroxide for 10 min appears to be as an easy, effective and inexpensive

method that can improve the clinical performance of methacrylate resin-based glass fibre posts.

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