The effect of air-blowing on the µTBS to dentin of four different adhesive systems

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Summary

Purpose: To evaluate the technique sensitivity of four different adhesive systems using different air blowing pressure.

Methods: Four adhesive systems were employed: Clearfil SE Bond [SE] (Kuraray, Japan), G-Bond [GB] (GC Corporation, Japan), Adper Prompt L-Pop [LP] (3M ESPE, USA) and an experimental adhesive, SSB-200 [SSB] (Kuraray, Japan). Twenty-four extracted molars were used. After grinding the coronal enamel surface, the teeth were divided into two equal groups. The first group's teeth were randomly assigned for bonding with the different adhesives using gentle air blowing (g). For the teeth of the second group, the four adhesive systems were applied using strong air blowing (s). After storage overnight in 37°C water, the bonded specimens were sectioned into sticks (1mm x 1mm wide) which were subjected to microtensile bond strength testing (μTBS) at a crosshead speed of 1mm/min. The load at failure of each specimen was recorded and the data were analyzed by one-way ANOVA and Tukey HSD tests. The surfaces of the fractured specimens were observed using SEM to determine the failure mode.

Results: The results of the μTBS test showed that the highest bond strengths tended to be with SE for both gentle and strong air blowing, and the significantly lowest for SSB with strong air streaming. Comparing the two techniques, significant differences were noted only for SSB-200 ($p<0.05$). For each material, the SEM evaluation did not show distinct differences in the nature of the fractures between the two techniques, except for SSB-200.

Conclusions: The adhesives tested are not technique sensitive, except SSB-200, with regards to the air blowing step.

INTRODUCTION

Adhesion of resin composite to the tooth substance is required to provide retention, reduce microleakage and improve marginal adaptation. Since its introduction, the enamel etch technique has provided an ideal surface for reliable bonding performance using adhesive resins.[1] However, bonding to dentine has been less reliable due to the characteristics of the dentine substrate, including high organic content,[2] tubular structure variations,[3] the presence of outward fluid movement,[4] dentine depth,[5] sclerosis[6] and caries.[7,8,9] Bonding of resin composite to dentine is now possible through partial demineralization of the dentine surface, followed by infiltration of the exposed collagen fibrils with hydrophilic resins to obtain the hybrid layer.[10,11]

Different adhesives systems are commercially available and they can be divided into two broad categories: the systems that use “total-etching” with 35-40% phosphoric acid, followed by the application of a primer and an adhesive (in one or two different solutions); and the “self-etching” systems in which the prior etching step is omitted. The self-etching products may have the priming and bonding steps combined (1-step systems) or they may require an additional step (2-step adhesives). For all these systems, adherence to the manufacturer’s instructions is implied for successful bonding to dentine. Some researchers have found that errors in application could result in lower bond strengths.[12]

Different authors have investigated the operator variability of these adhesive systems and have found that technique sensitivity is one of the most important variables in the use of these kinds of materials.[13,14,15] Frankenberger[16] showed that one of the most critical steps during the application of "total-etching" systems is the control of the dentine's moisture after the rinsing step. Nowadays, with the introduction of "self-etching" systems involving better standardized procedures without water rinsing or dentine moisture retention, this problem should theoretically be eliminated.[17] In particular, the newest "all-in-one" systems should be less technique sensitive due to the reduction of their application to only one step.[18] However, other variables related to their application could perhaps influence the bond strength of these adhesives, such as the air blowing step. Previous research has reported that manufacturers' instructions are often not strictly adhered to and this may affect the bond strength of the adhesives.[19] Therefore, an adhesive whose bond strength is not heavily reliant on the technique sensitivity related to its application, may perform better in the clinical situation.

Air blowing is important to eliminate substances that could influence polymerization and to ensure a good distribution of the adhesive on the dentine surface.[20] The duration of air blowing and the pressure of the air stream are different for each product. It is not known whether deviations from the suggested protocols for air blowing affect bond strength values.

The purpose of this study was to evaluate the effect of technique sensitivity on μTBS of four different "self-etching" (3 commercial and 1 experimental) adhesives, focussing on the air blowing step. The null hypothesis tested was that μTBS was

not affected by air blowing.

MATERIALS AND METHODS

Twenty-four non-carious human molars, stored at 4°C in an aqueous solution of 0.5% chloramine-T, were used for this study according to local institutional guidelines. The occlusal third of the molar crowns was removed by means of an Isomet diamond saw (Isomet 1000, Buehler, Lake Bluff, IL, USA) to expose the dentine surface. A standard smear layer was produced by treating the surface with 600-grit silicon-carbide paper. All specimens were randomly divided into two groups of twelve teeth each. Four self-etching adhesive systems were employed for this experiment: one 2-step system, SE Bond [SE] (Kuraray Medical Inc, Okayama, Japan) and three all-in-one systems: G-Bond [GB] (GC Corporation, Tokyo, Japan), Adper Prompt L-Pop [LP] (3M ESPE, St Paul, MN, USA), and an experimental adhesive SSB-200 [SSB] (Kuraray Medical Inc, Okayama, Japan). The chemical formulations of the four adhesives are listed in Table 1. The respective manufacturer's instructions for usage of the four adhesives employed are listed in Table 2.

In order to evaluate the technique sensitivity, the teeth of the first group were prepared in accordance with the times suggested by the individual manufacturer's instructions and using gentle air blowing, while for the teeth of the second group, strong air blowing was employed. In each case, the air stream was generated by a commercially available canister of compressed contamination-free air for 3 seconds (EM Clean spray, Nisshin EM Co, Tokyo, Japan) at a distance of 2 cm from the specimen. The strong air stream was at a pressure of 0.68 MPa while the gentle air blowing occurred approximately at

0.12 MPa. Three teeth per adhesive per technique were prepared. All procedures were carried out by one operator. All surfaces were built up with resin composite (Clearfil AP-X, Kuraray Medical Inc, Okayama, Japan) in increments to a height of 5 mm. Each incremental layer was light cured for 40 seconds. After 24h storage in water at 37°C, approximately six sticks (1mm x 1mm wide) were obtained from each sample using an Isomet diamond saw. Specimens were then fixed to a Ciucchi's jig [21] with cyanoacrylate glue (Model Repair II Blue, Dentsply-Sankin, Otahara, Japan) and subjected to a tensile force at a crosshead speed of 1 mm/min in a desk-top testing apparatus (EZ test, Shimadzu, Kyoto, Japan) until failure occurred. The μTBS was expressed in MPa, dividing the applied force (N) at the time of fracture by the bonded area (mm^2). Data were evaluated with one-way ANOVA to detect any statistical differences. The Tukey HSD test was used to analyze differences between the two techniques at a significance level of $p=0.05$.

Both halves of the fractured specimens were observed using an optical light microscope (LG-PS2, Olympus Co, Tokyo, Japan) at X20 magnification, and then sputter coated before being observed in a FE-SEM (S-4000, Hitachi, Tokyo, Japan) at an accelerating voltage of 10 kV, to determine the failure mode. All observations were conducted by one person.

RESULTS

Microtensile Bond Strength Test

A total of 137 specimens from 24 teeth were available for microtensile testing. There were pretesting failures in 2 samples of LP(s) and in 6 samples of SSB(s). These pretesting failures were considered as 0 MPa and were included in the analysis. The mean μTBS for the data in MPa are presented in Table 3 and displayed in Fig. 1. Overall, SE tended to have the highest bond strengths for both gentle and strong air blowing, and the statistically lowest bond strengths were for SSB with strong air streaming ($p<0.05$).

Statistical analysis using the Tukey HSD test for each material showed a significant difference between the two techniques only for SSB-200.

SEM Analysis

The vast majority of the specimens showed visually adhesive failures (at x20). Observation by SEM at low magnification (x50) demonstrated the same pattern in all groups. However, at higher magnifications differences began to appear in the mode of failure; the SEM images showed different failure patterns at the interface of the adhesives employed.

The majority of the SE samples, with both gentle and strong air, showed fractures within the adhesive layer (Fig. 2a). In the rest of the samples, the fracture occurred primarily within the hybrid layer and the adhesive (Fig. 2b)

G-Bond showed fractures at the interface between the dentine and the adhesive as well as cohesive fractures within the adhesive layer in the same sample and this was observed in both groups (Fig. 3a). Many “blisters” were noted in the

adhesive layer, irrespective of the technique employed. However, the number (2.15 ± 0.34 μm^2) and size (range: 0.07- 1.85 μm) of the “blisters” were greater in the gentle air-blowed specimens compared with those that were subjected to strong air blowing (number: 1.80 ± 0.21 μm^2; size: 0.07- 1.70 μm) (Fig. 3b).

Prompt L-Pop samples treated with gentle air blowing showed mixed fractures. Generally, the surfaces presented a portion in which the failure occurred between the dentine and the adhesive layer, and another part with a cohesive fracture within the adhesive (Fig 4a). The majority of the samples prepared with strong air blowing showed areas with collagen fibrils and resin tags in the tubules while other areas displayed the hybrid layer (Fig 4b).

SSB-200 showed significant morphological differences between the teeth prepared with the two techniques. For the samples treated with gentle air, the fractures occurred between the adhesive layer and the resin composite and/or cohesively within the adhesive (Fig 5a). The teeth prepared with strong air blowing presented fractures within the adhesive layer only. Higher magnification of the adhesive layer revealed the presence of many voids (Fig 5b).

DISCUSSION

Generally, current interest in dentine bonding research is focused on reducing the number of application steps in the bonding procedure and reducing the technique sensitivity as well as operator variability. The all-in-one adhesives have reduced the number of steps involved. There are two kinds of all-in-one systems, the two-bottle adhesives and the one-bottle systems. Prompt L-Pop falls into the category of two-bottle systems while G-Bond and SSB-200 are one-bottle adhesives. In contrast, SE Bond is a two-bottle two-step adhesive. Although the application procedure has been simplified by the all-in-one adhesives, their technique sensitivity remains unknown.

While Miyazaki *et al*[20] have shown the importance of the primer application method in the resultant bond strength of adhesives, the specific effect of the air blowing step on the all-in-one systems has not been previously investigated. This study assessed the effect of gentle air blowing and strong air on the bond strengths of these systems compared to a two-step adhesive. The two-step system used in this study, SE Bond, has consistently demonstrated high bond strengths in other studies[22, 23, 24] and hence was used in this investigation.

SE Bond showed the highest bond strengths regardless of gentle and strong air blowing during the application of the primer. The fact that most of the fractures occurred within the adhesive layer suggests that this system is less technique sensitive to the air blowing step. The bond strengths were not statistically influenced by air-blowing. This is perhaps due to the fact that application of the bonding resin may have compensated for the strong air blowing. The components of the bonding resin in SE Bond (10-MDP, Bis-GMA and HEMA)

may have a good affinity to primed and a strongly air blown dentin surface in the same way as to a gentler blown surface.

Although the instructions for G-Bond state that strong air blowing is required, this is not borne out by the results of this study in terms of the μTBS. Moreover, the fracture patterns for both strong and gentle air blowing were similar. An unique feature noted with this system was the presence of “blisters” within the adhesive. The number and size of these blisters were greater in the gentle air-blowed specimens compared to the strong air-blowed samples. An important difference between G-Bond and the other adhesives used was that it does not contain hydrophilic monomers such as HEMA. HEMA is soluble in water, acetone and alcohol.[25] HEMA is also an organic material that has an affinity to hydrophobic monomers. Hence, it can be a useful medium for the hydrophilic and hydrophobic components. On the other hand, HEMA creates a hydrogel within the hybrid layer and adhesive resin in some cases. [26] The hydrogel may provide a channel for water permeation that has the potential to affect the durability of bonds, especially when poly-HEMAs of low molecular weight are created.

G-Bond contains acetone as a solvent. However, as it does not contain HEMA, it is speculated that evaporation of the acetone in G-Bond could result in phase separation of the components. This may be the reason for the blister formation noted in this adhesive. Furthermore, the blisters were greater in the samples that were subjected to gentle air blowing. While strong air blowing may prevent pooling of the adhesive on the substrate surface, gentle air blowing may encourage pooling. The combination of evaporation of acetone and pooling may result in greater phase separation. Phase separation has been reported by other

researchers[27] as has blister formation. [26] The long-term effects of this are unknown.

It is not apparent as to why there was no difference between strong and gentle air blowing with G-Bond. Blister formation in gentle air-blowed samples appeared greater in number and the bond strengths were lower than in gentle air-blowing. This may be because the large numbers of defects could have acted as the origins of the fractures. However, the mechanically properties of the cured resin in both groups may have been similar which led to no statistical difference in the results. Further TEM observation and mechanical testing of the adhesive resin are needed for future work.

Prompt L-Pop did not show significant differences in μTBS between the gentle and strong air-blowed samples. However, pretesting failures occurred in the strong air-blowed group. SEM examination of the samples in both groups showed differences in the fractured surface. The typical fracture pattern in the gentle air-blowed group was mixed, while strong air blowing resulted in interfacial failure. This latter type of failure is usually suggestive of a weak bond between the adhesive and the substrate. This is probably also the reason for the pretesting failures obtained in the strong air blowing group. Strong air blowing may have caused over-removal of the adhesive resin causing incomplete enveloping of the collagen fibrils (Fig 4b) leading to early failure in some samples and adhesive failure during testing in others. The lack of statistical difference between the two groups may be partly due to the large standard deviation of the data in both groups. However, the bond strength of strong air-blowed samples tended to be lower compared to the gentle air-blowed group.

The experimental adhesive, SSB-200, showed significantly lower bond strengths in the strong air-blowed group than the gentle air group. The strong air group demonstrated the lowest bond strengths in the study. In addition, several pretesting failures were noted in the strong air group. These differences between both groups were confirmed by the morphological findings. The gentle air-blowed group showed more complex failures (at different planes), while those in the strong air group consistently failed within the adhesive. Figure 5b shows the presence of many voids in the adhesive. The authors speculate that these voids are pockets of air which could be stress raisers within the cured adhesive resin during testing. This may be a big reason for the poor adhesion in this group. It is speculated that when strong air-blowing is used, water and solvents are evaporated quickly resulting in a viscous resinous material with entrapped air bubbles, remaining on the dentin surface. This would lead to weaker mechanical properties, resulting in lower bond strengths.

Since the introduction of self-etching primers, gentle air blowing was generally required for removing the excess primer solution. It is commonly recommended by the manufacturers that gentle blowing should be performed in order to achieve higher bond strengths. However, the instructions for G-Bond represent a departure from the usual protocol. Inspite of this, there were still the presence of blisters within the adhesive. Clinicians are usually accustomed to gentle air blowing when using the self etching systems. Gentle air blowing reduces the possibility of dispersal of the components of the adhesive into or out of the oral environment. This is particularly important as many adhesives contain HEMA and organic components. In this study, strong air blowing did not improve the

bond strengths of the adhesives tested; in fact, this had an adverse effect on one of them (SSB-200). It also resulted in pretesting failures in two of the adhesives (LP and SSB). Hence, the use of strong air blowing with these adhesives is not recommended.

The null hypothesis that μTBS was not affected by air blowing was accepted for SE Bond, G-Bond and Prompt L-Pop, but rejected in the case of SSB-200. However, pretesting failures were observed in the Prompt L-Pop and SSB-200 groups. In conclusion, the adhesives used were not technique sensitive in terms of the air blowing step, except for Prompt L-Pop and the experimental adhesive, SSB-200.

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LEGENDS

Figure 1: Mean μTBS and the SDs in MPa of all specimen groups.

Figure 2a: SEM photomicrograph of the fractured surface on the dentine side of a sample of Clearfil SE Bond, typical of both gentle and strong air blowing procedures. The fracture occurred within the adhesive layer.

Figure 2b: The fractured surface (dentine side) of Clearfil SE Bond showing the hybrid layer on dentine (H) and the bulk of the adhesive (A).

Figure 3a: Photomicrograph of fractured surface of G-Bond (strong air) showing the dentine (D), cohesive failure within the adhesive layer (A) and the interface between the adhesive and resin composite (I). Resin tags are present in the tubules (arrowed) and blisters were noted within the adhesive layer.

Figure 3b: SEM photomicrograph of a G-Bond sample treated with gentle air blowing showing the presence of blisters within the adhesive. In this particular sample, the failure also occurred at the interface (I) between the adhesive (A) and the resin composite.

Figure 4a: Fractured surface of a Prompt L-Pop sample prepared with gentle air blowing showing a portion in which the failure occurred between the dentine (D) and the adhesive layer, and another part with a cohesive fracture within the adhesive (A). Remnants of the

resin composite (C) are present in this specimen.

Figure 4b: SEM photomicrograph of a typical Prompt L-Pop specimen prepared with strong air, showing areas with collagen fibrils and resin tags (arrowed) in the tubules while other areas display the hybrid layer (H) on the dentine.

Figure 5a: The fractured surface of an SSB-200 specimen (gentle air) showing a part with the fracture at the interface (I) between the adhesive layer and the resin composite and another area with cohesive fracture within the adhesive (A).

Figure 5b: SEM photomicrograph of a typical SSB-200 sample (strong air) at high magnification showing the presence of voids (V) and fillers (F) within the adhesive.

TABLES

Product Name	Manufacturer	Composition
SE Bond Lot: 011370	Kuraray Medical Inc, Okayama, Japan	Primer: 10-MDP, HEMA, hydrophilic dimethacrylate, photoinitiator, water Bond: 10-MDP, Bis-GMA, HEMA, hydrophilic dimethacrylate, microfiller
G-Bond Lot: 0406161	GC Corporation, Tokyo, Japan	4-MET, Phosphate monomer, UDMA, water, acetone, silica filler, photo initiator
Prompt L-Pop Lot: 180505	3M ESPE, St Paul, MN, USA	Liquid 1: Methacrylated phosphoric esters, Bis-GMA, camphoroquinone, stabilizers Liquid 2: Water, HEMA, polyalkenoic acid, stabilizers
SSB-200 Lot: 040219	Kuraray Medical Inc, Okayama, Japan	10-MDP, HEMA, water, ethanol, dimethacrilate, photoinitiator, filler

Table 1: Chemical formulations of the four adhesive systems used.

Adhesive	Manufacturer's instructions
SE Bond	- Apply the primer for 20s - **Gentle air blowing** - Apply the adhesive for 10s - **Gentle air blowing** - Light cure for 10s
G-Bond	- Apply the adhesive for 10s - **Strong air blowing** - Light cure for 10s
Prompt L-Pop	- Activate the blister - Apply the adhesive massaging for 15s - **Gentle air blowing** - Apply a second coat without massaging - **Gentle air blowing** - Light cure for 10s
SSB-200	- Apply the adhesive for 20s - **Gentle air blowing** - Light cure for 10s

Table 2: Manufacturers' instructions for the four adhesive systems.
Note: only for G-Bond, the manufacturer's instructions suggest "strong air blowing".

Adhesive	Mean	SD	N	Pretesting failures
SE Bond (g)	74.86[a]	20.74	19	0
SE Bond (s)	68.59[a,b]	14.90	16	0
G-Bond (g)	44.60[c,d]	11.13	20	0
G-Bond (s)	48.77[c]	12.21	18	0
Prompt L-Pop (g)	43.92[c,d]	16.96	16	0
Prompt L-Pop (s)	31.01[d]	18.46	20	2
SSB-200 (g)	56.29[b,c,*]	12.35	17	0
SSB-200 (s)	12.32[e,*]	11.83	19	6

Table 3: Mean μTBS (MPa) for all specimen groups.

g=gentle air; s=strong air.

N refers to the total number of specimens including the pretesting failures.

* denotes a significant difference between the two groups.

The same superscripts denote no statistical difference.

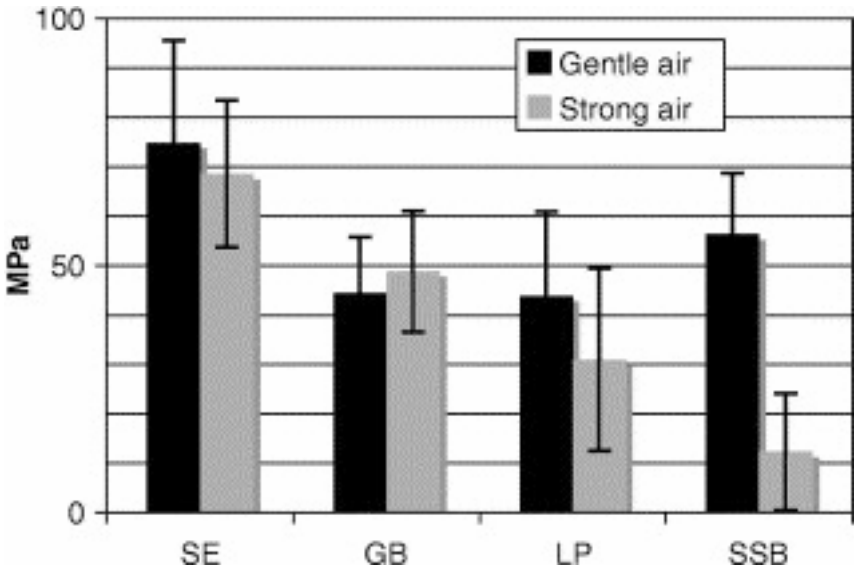

Figure 1

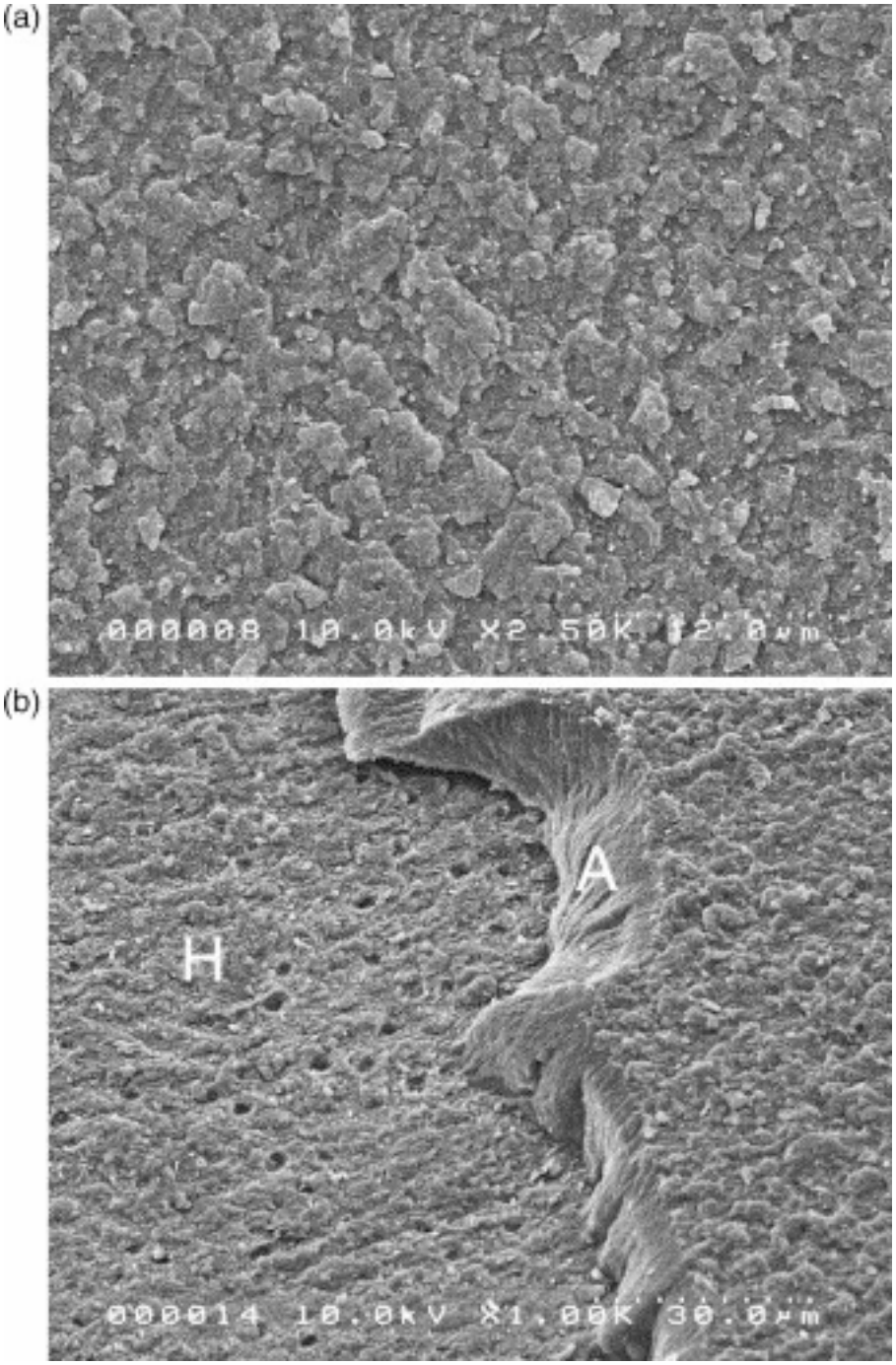

Figure 2

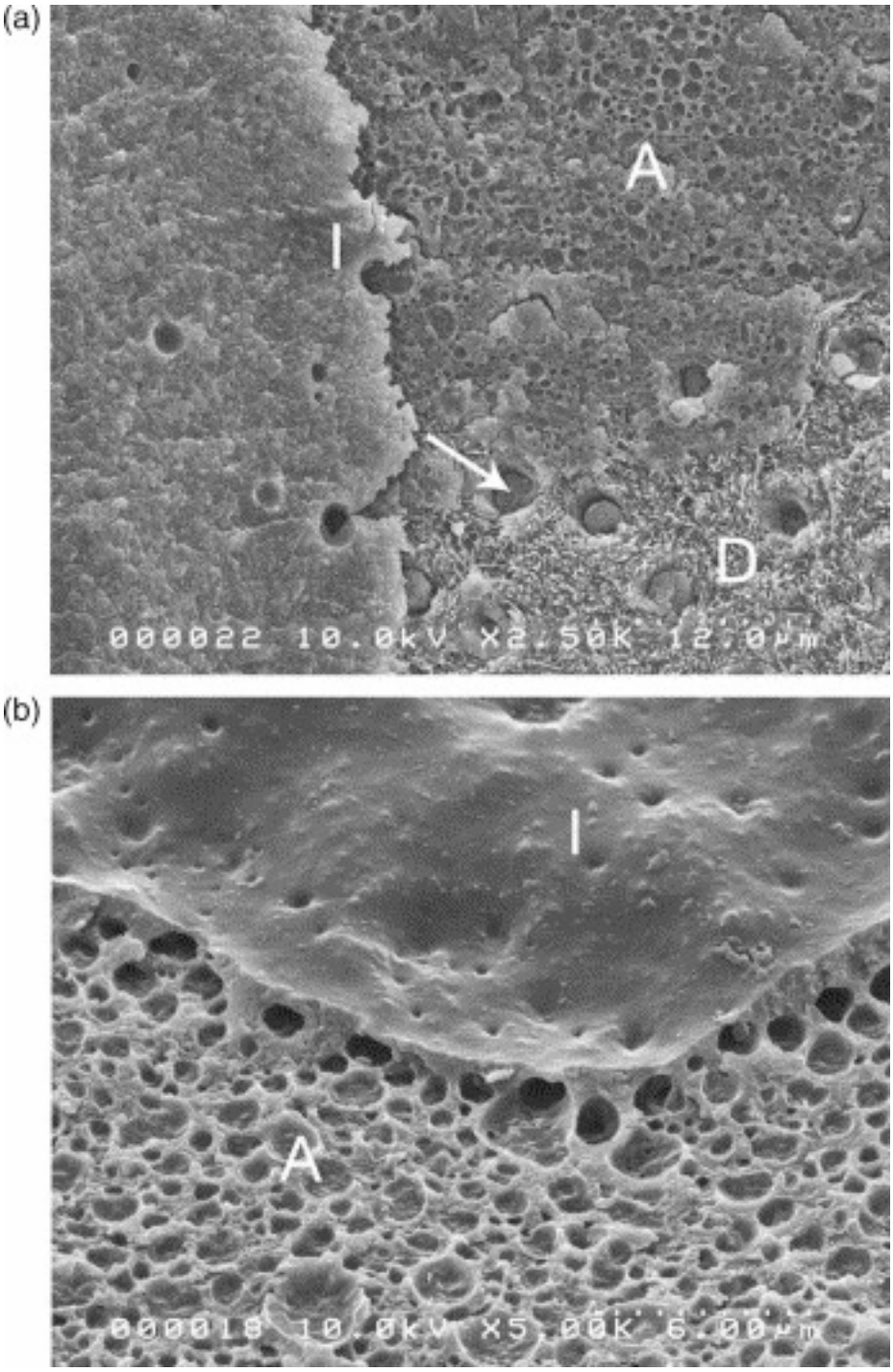

Figure 3

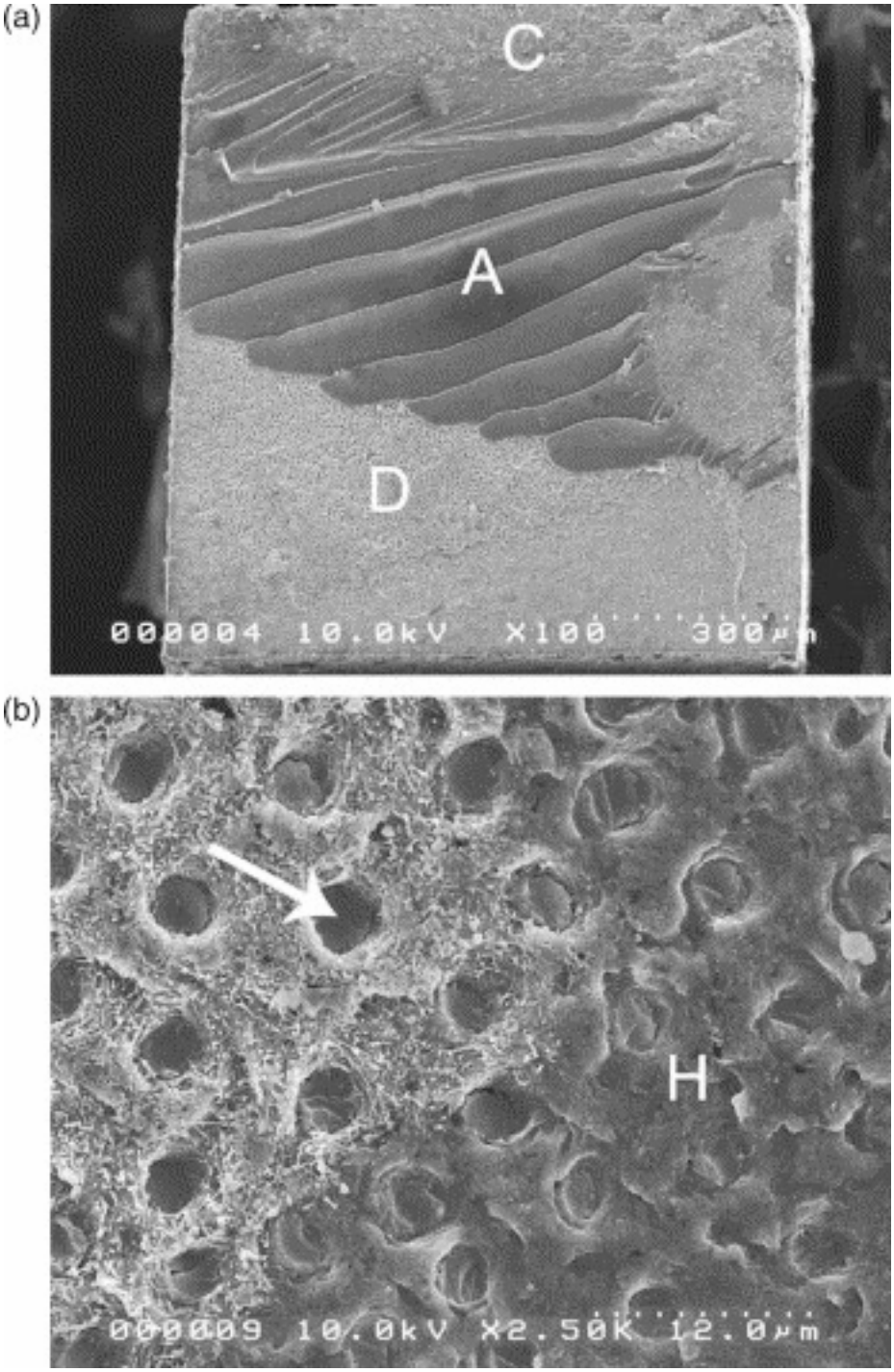

Figure 4

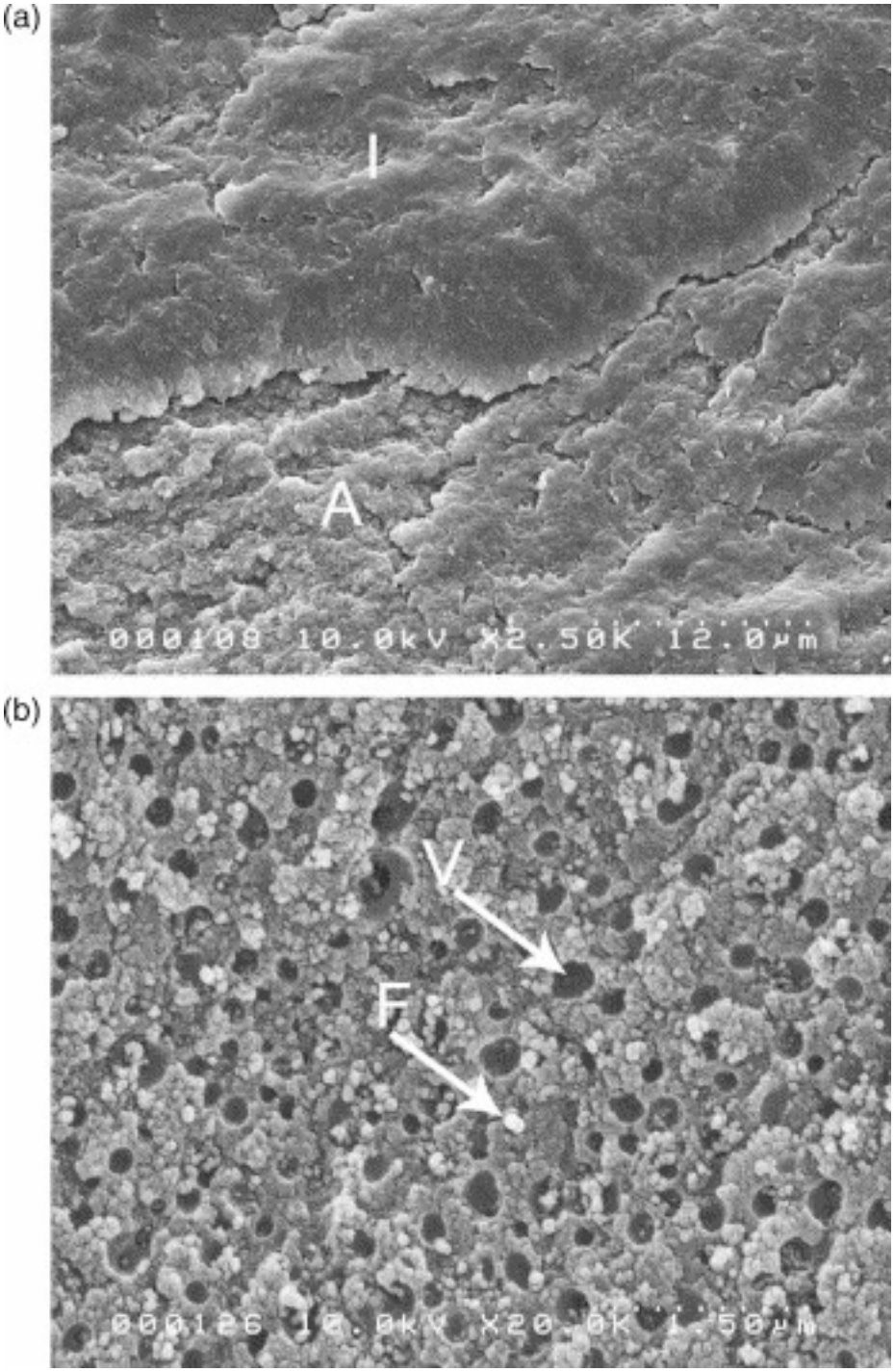

Figure 5