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Dentin bonding as a function of dentin structure

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The availability of new scientific information about the etiology, diagnosis, and treatment of carious lesions and the introduction of new adhesive restorative materials have substantially reduced the need for extensive tooth preparations [1]. Adhesive composite restorations are now used to replace carious dental tissues, to restore fractured teeth, to replace missing enamel or dentin in the cervical areas of teeth, or to change the shape and shade of anterior teeth. With improvements in materials, these indications have progressively shifted from anterior to posterior teeth. The capability of clinicians to bond restorative materials to enamel and dentin has fundamentally changed concepts of cavity preparation, orthodontic treatment, caries prevention, and cementation of fixed prostheses, including prefabricated zirconia and carbon fiber posts.

Modern adhesive techniques allow dentists to confine operative procedures to removal of diseased tooth tissue, thus preserving sound tissue. The short lifetime of restorations, which are frequently assessed by methods not based on clinical evidence, [2,3] requires clinicians to replace restorations frequently. [4–6] Each time the restoration is replaced, an additional part of remaining sound tooth structure is inevitably removed, and consequently, an enlarged and more complex restoration is needed. [7] Extending the restoration lifetime is therefore one of the primary goals of research being now carried out worldwide in dental materials research.

The idea of applying phosphoric acid on the enamel surface was based on the observation that the industrial application of phosphoric acid to metal surfaces resulted in better adhesion of paints and resin coatings. [8] Since

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Buonocore's introduction of the acid-etch technique, many dental researchers have attempted to achieve methods for reliable and durable adhesion between resins and tooth structure. Acid etching transforms the smooth enamel into a very irregular surface (Fig. 1). After rinsing off the etchant with water and drying the enamel surface with air, a fluid resin is applied on the enamel surface. This resin penetrates into the subsurface, drawn by capillary action (Fig. 2). Monomers in the fluid resin polymerize and become interlocked with the enamel surface. The formation of resin microtags (Figs. 3 and 4) within the enamel structure is the fundamental mechanism of adhesion of resin to enamel. [9,10]

As opposed to enamel, which is composed of more than 90% of hydroxyapatite and can be dried easily, dentin is an intrinsically wet organic tissue penetrated by a tubular maze (Fig. 5) containing the odontoblastic process, which communicates with the pulp. The density of the tubules by unit area is greater close to the pulp than near the dentin-enamel junction (Fig. 6). [11] The dynamic nature of dentin as a substrate is responsible for inconsistent bond strengths and marginal leakage, which still occur with all resin-based adhesives. [12]

Whenever tooth structure is prepared with a bur or other instrument, residual organic and inorganic components form a "smear layer" of debris on the surface. [13,14] The smear layer fills the entrance of dentin tubules to form smear plugs (Fig. 7), which decrease dentin permeability by up to 86%. [15] Submicron porosity of the smear plug still allows for flow of dentinal fluid. [16] Although the smear layer acts as a "diffusion barrier" that decreases the permeability of dentin, [15] it also can be considered an obstacle that must be removed so that resin can be bonded to the dentin substrate. Based on that consideration, several dentin bonding techniques have been introduced in the past 40 years. Current dentin bonding systems are generally grouped into two categories in terms of how they interact with the dentin smear layer (Tables 1 and 2):

- Dentin adhesives that treat the dentin and enamel surface with a nonrinsing solution of acidic monomers in water (also known as *self-etching primers*). These bonding systems do not remove the smear layer (Fig. 8) but make it permeable to the monomers subsequently applied. [12]
- Dentin adhesives that include an acid gel to treat the dentin and enamel (generally 30–40% phosphoric acid) for 15 to 30 seconds. Opening of the dentinal tubules and removal of the dentin smear layer by acid etching [17] (Fig. 9) have led to significant improvements in the in-vitro bond strengths of resins to dentin. [13,18,19]

The ideal dentin substrate

Detailed knowledge of the structure of human dentin is essential to evaluate and improve adhesive restorative systems. The specificity of dentin

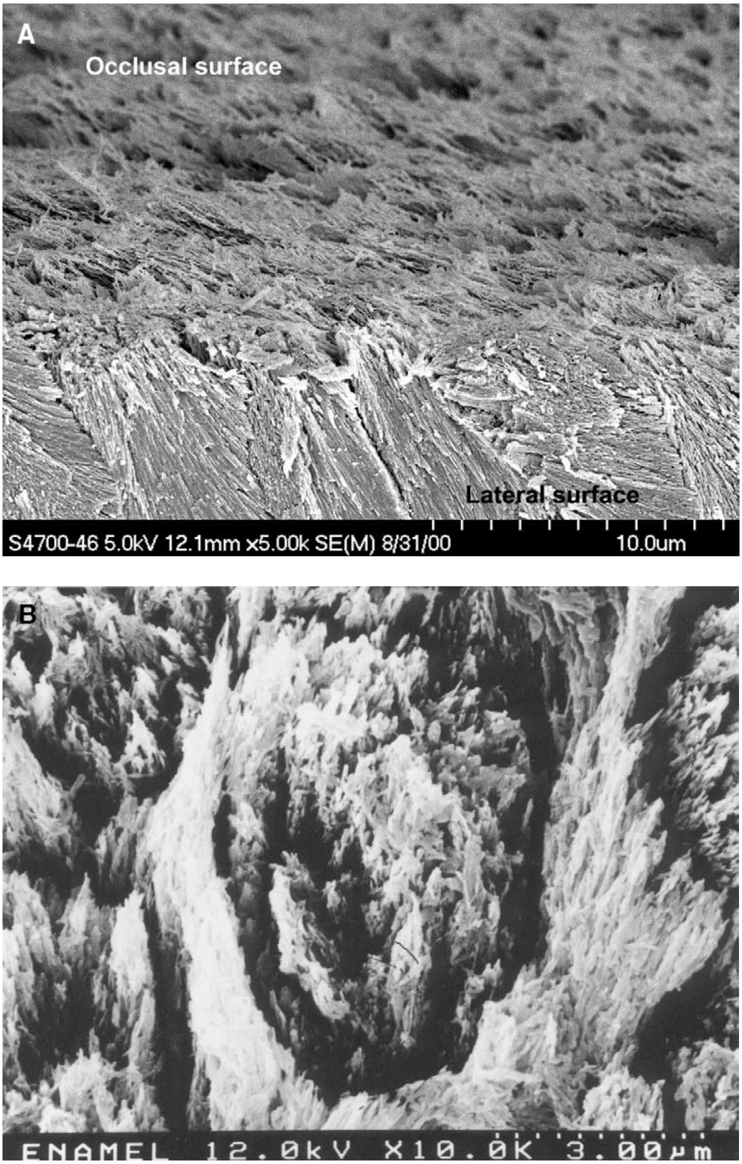

Fig. 1. (A) Surface roughness of enamel etched for 15 seconds with 35% phosphoric acid. (B) Enamel prism after etching with 35% phosphoric acid. Note the micro-porosites.

histology is the result of the interaction between the ectodermal and ectomesenchymal components of the tooth germ that induces odontoblast differentiation and dentinogenesis. As a result of the production of dentin, the odontoblasts leave a track throughout the dentin tissues, forming the

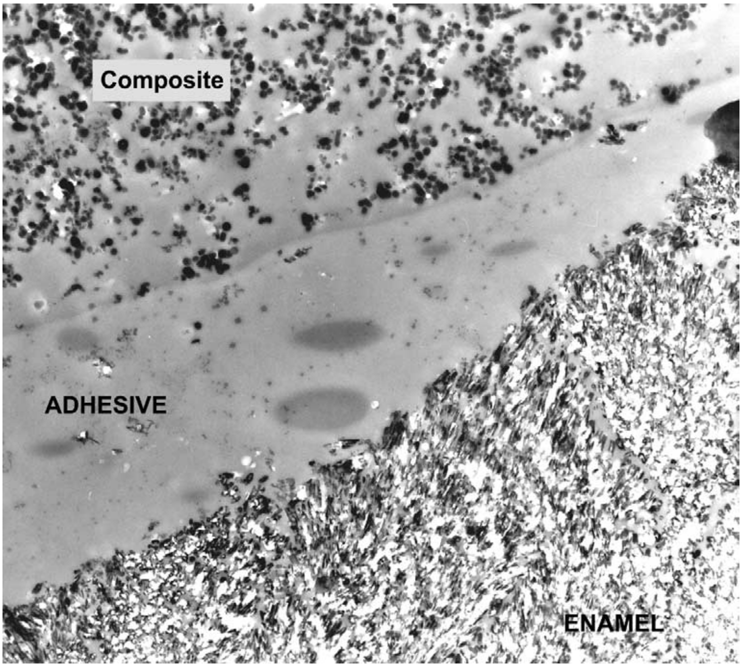

Fig. 2. TEM image of enamel-composite interface. The adhesive penetrated between the enamel crystallites to form micro-tags (asterisk). Original magnification = ×10,000.

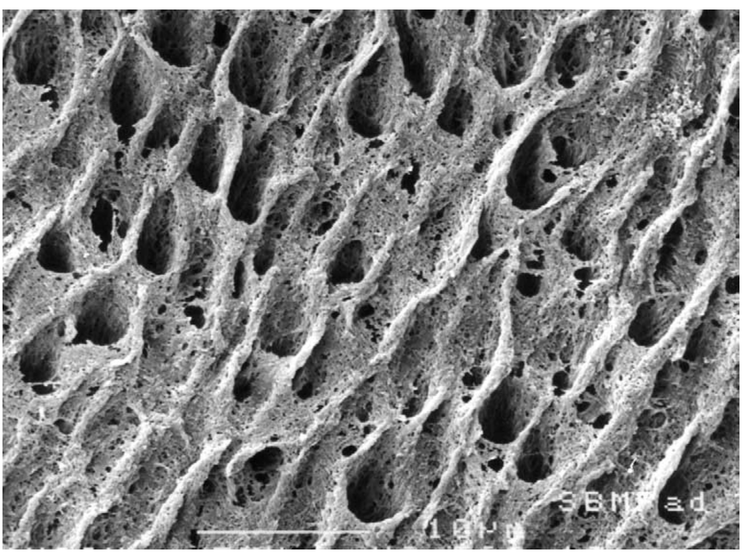

Fig. 3. Enamel etching pattern with Scotchbond Multi-Purpose (3M ESPE) adhesive after dissolution with 6N HCL for 30 seconds.

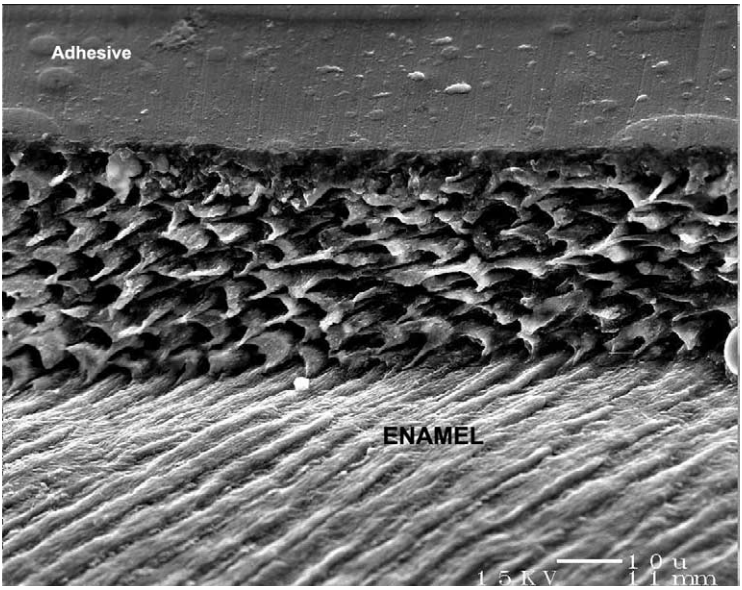

Fig. 4. Enamel fitting surface of Single Bond (3M ESPE) upon dissolution with 6N HCL for 30 seconds. Specimen tilted at 45°. Note the adhesive tags.

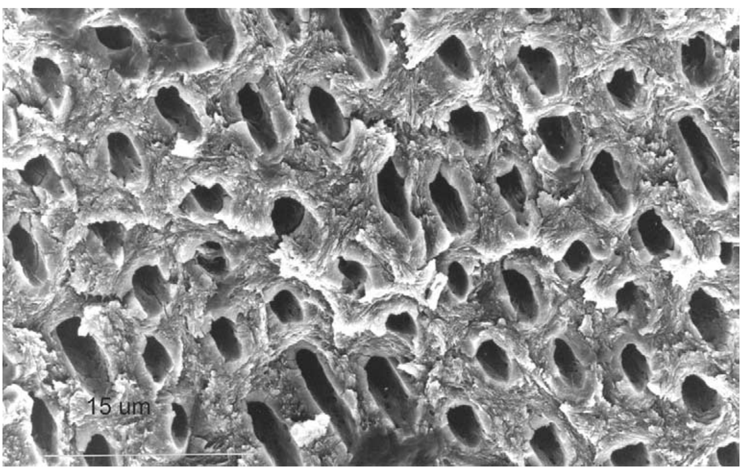

Fig. 5. Dentin tubules in middle dentin. Original magnification = ×2,500.

tubules. [20] The dentin tubules extend from the dentin-enamel junction (or cement) to the pulp chamber, presenting different density and orientation in distinct locations of the tooth. [11,21] Dentinal tubules enclose cellular extensions of the odontoblast, and therefore are in direct communication with the pulp. Inside the tubule lumen, other fibrous organic structures that

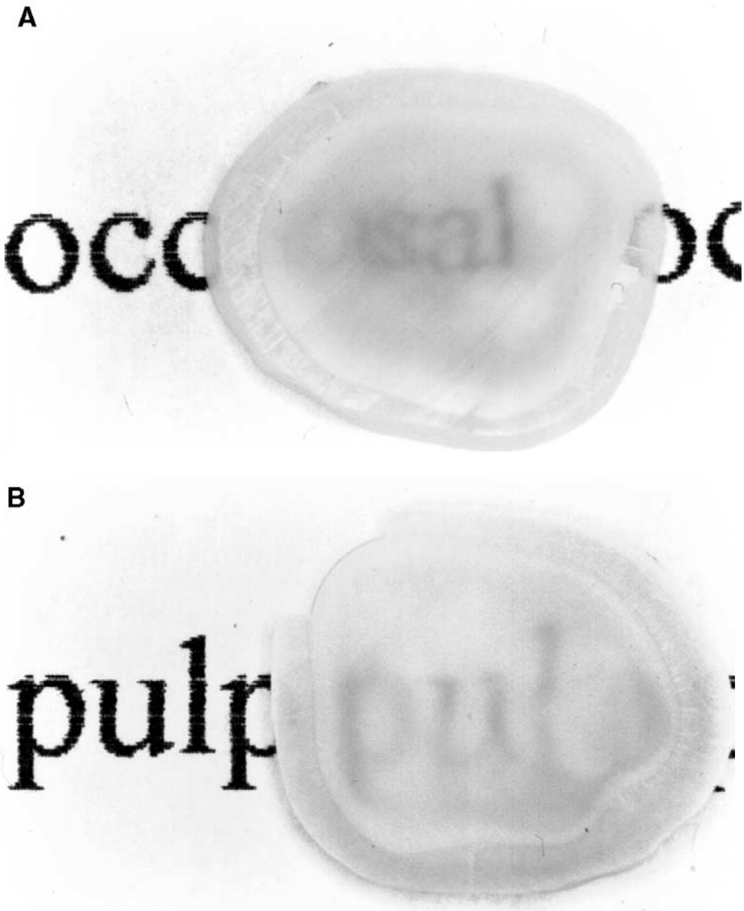

Fig. 6. (A) In the same dentin disk, there are fewer tubule apertures on the occlusal side than on the pulpal side. Letter size is reduced in contact with the occlusal surface. (B) Letters are enlarged when in contact with the pulpal surface.

substantially decrease the functional radius of the tubule can be observed, such as the lamina limitans.

The tubular structure of dentin is responsible for its intrinsic hydration owing to communication with the pulp tissue, which is under vascular pressure. [22] The scaffold of all the tubules and intertubular dentin is represented by the collagen fibrils produced by the odontoblasts. The precipitation of mineral substance on the collagen fibrils during dentinogenesis results in the final mineralized structure. Mineral, in the form of carbonate-rich apatite, constitutes approximately 50% of the dentin volume. [23]

The characteristic collagen banding is formed by a longitudinal staggering of the molecules that involves about one quarter of the length of the

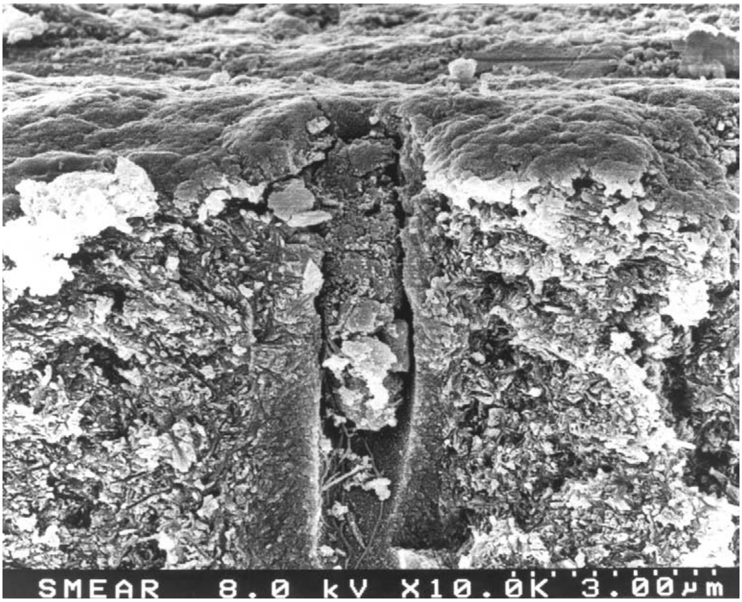

Fig. 7. Smear layer and smear plug upon using a diamond bur under water spray.

fibril monomer, leaving a space between the extremity of one collagen triple helix and the beginning of the next. This "hole" has been considered a site for accumulation of hydroxyapatite crystals inside the collagen molecule. [24] Collagen banding also has been reported in the peritubular region of

Table 1
Current types of dental adhesives

Type of adhesive	Acid	Primer	Fluid resin
Self-etching multibottle	One or two bottles; one application, no rinsing	Same as acid	One bottle; one coat
Self-etching "all-in-one"	Sequentially activated reservoirs for Prompt L-Pop; two bottles for One-Up Bond (mix one drop from each bottle). Several coats [42], no rinsing.	Same as acid	Same as acid
Total-etch multibottle (fourth generation)	Phosphoric acid; 15 sec, rinse, leave moist	One or two bottles; one to five coats	One bottle; one coat
Total-etch one bottle (fifth generation)	Phosphoric acid; 15 sec, rinse, leave moist	One bottle; One, two, or three+ coats	Same as primer

(*Adapted from* Perdigão J, Frankenberger R, Rosa BT, Breschi L. New trends in dentin/enamel adhesion. Am J Dent 2000;13:25D–30D; with permission.)

Table 2
Advantages and disadvantages of current dental adhesives

Type of dentin adhesive	Advantages	Disadvantages
Self-etching multicomponent (Clearfil SE Bond, Clearfil Liner, Bond 2V, and Experimental ABF [Kuraray Co., Osaka, Japan], Etch & Prime 3.0 [Degussa, AG, Düsseldorf, Germany])	• No rinsing, quick application • Less postoperative sensitivity than total-etch adhesives • Results of clinical studies support use on dentin, but may not be effective on sclerotic dentin • Bonds well to dentin etched with phosphoric acid	• Clinically, may result in enamel microleakage due to a deficient enamel etch • Slight degradation of the hybrid layer over 1 year, in vivo
Self-etching "all in one" (Prompt L-Pop [3M ESPE], One-Up Bond [Tokuyama Co., Tokyo, Japan])	• No rinsing, very quick application • Results in an enamel etch pattern similar to that of phosphoric acid if enamel is instrumented • No bottles, disposable container (for Prompt L-Pop)	• Does not bond well to unprepared enamel • Has resulted in a wide range of bond strength values • Prompt L-Pop bonds better with compomers than with composites • One-Up Bond still requires mixing of two components with a brush • Needs multiple coats to bond effectively to dentin • Not indicated for indirect restorations
Total-etch multibottle (fourth generation) (All-Bond 2 [Bisco], EBS Multi [3M ESPE], OptiBond FL [Kerr Co.], Scotchbond Multi-Purpose [3M ESPE])	• Several research reports support its use on different substrates, including metals and porcelain • The highest dentin bond strengths among all dentin adhesives • Generally contain a dual-cure option for indirect restorations and bonded amalgams	• Multiple bottles make utilization cumbersome • Some bottles in the kit may never be used • Possibility of running out of primer A before primer B (or vice versa) • Because primer and adhesive resin are dispensed into the same plastic container, their sequential application may be inverted

Total-etch one bottle (fifth generation) (Bond 1 [Pentron Clinical Technologies LLC, Wallingford, CT], Dentastic Uno/Duo [Pulpdent Co., Watertown, MA], Excite [Ivoclar Vivadent, Schaan, Liechtenstein], Integra Bond [Premier Dental Products, King of Prussia, PA] One Step [Bisco], OptiBond SOLO Plus [Kerr Co.], PQ1 [Ultradent, South Jordan, UT], Prime & Bond NT [Dentsply]) Single Bond [3M ESPE]	• Laboratory research supports use on enamel and dentin • Clinical studies up to 3 years show positive results • The one-bottle concept makes it extremely user-friendly	• For some one-bottle adhesives, lower bond strengths than their multibottle adhesive counterparts • Acetone-based adhesives may lose their efficacy with constant use • Acetone-based adhesives may need more coats than those recommended by the manufacturer • Thick adhesives may pool easily around the preparation margin • Some one-bottle adhesives are not compatible with self-cured or dual-cured composites (build-up composites and resin luting cements)

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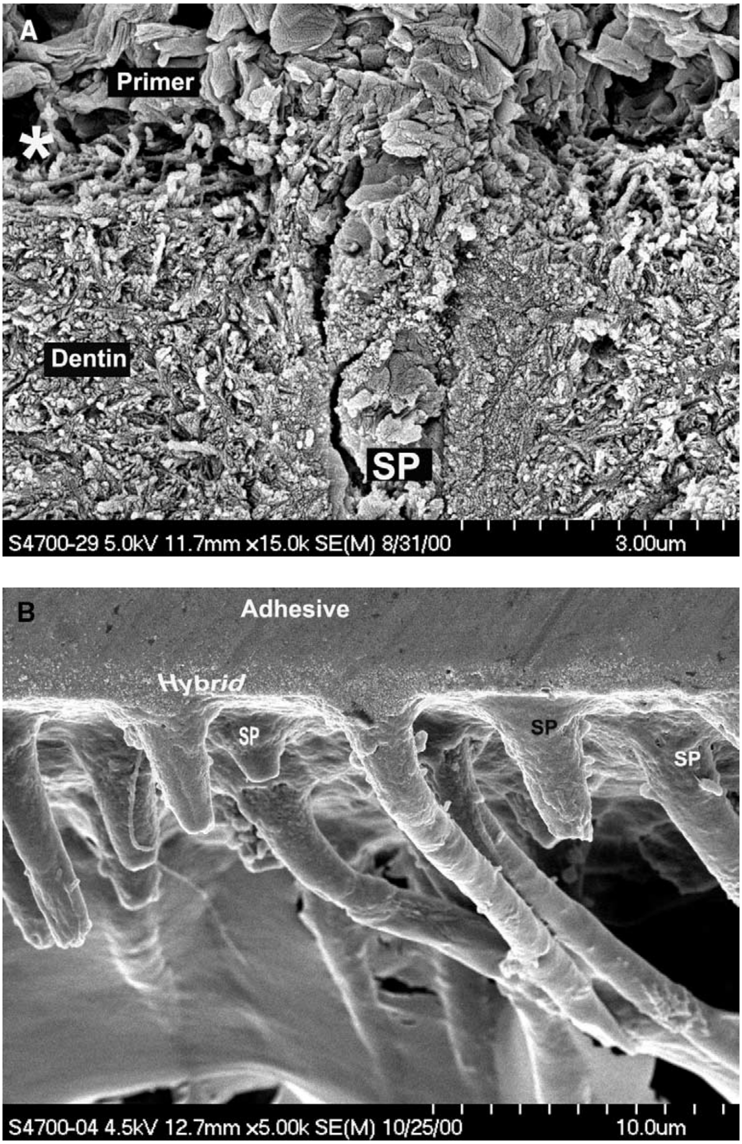

Fig. 8. (A) Resin-impregnated smear plug (**SP**) upon treatment with a self-etching primer. The demineralized dentin is marked with an asterisk. (B) Same self-etching material as in (A), but the adhesive has now been applied (dentin dissolved away). Note the smear plugs embedded in the resin tag necks (**SP**).

etched dentin at etching. [25] The lack of collagen banding in some areas of etched dentin may be caused by the aggressiveness of the etchant, because dentin collagen, after demineralization, is susceptible to proteolytic degradation. [26,27] Despite assumptions that etching dentin for 15 seconds might

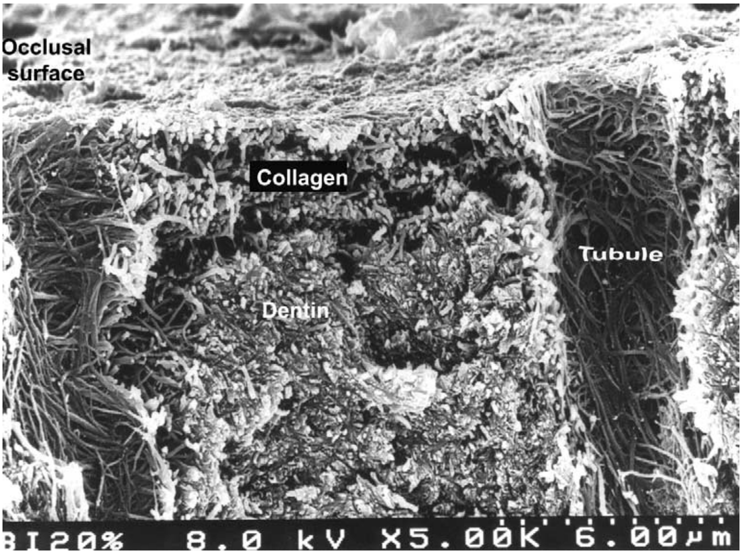

Fig. 9. Lateral view of dentin after etching with 20% phosphoric acid for 15 seconds. Note that the acid penetrated 4 µm into the intertubular zone.

cause collagen denaturation and compromise dentin bonding, it has never been substantiated nor has it been proven that changes in collagen ultrastructure are detrimental to the performance of total-etch dentin adhesives. In a recent immunocytochemical study, [28] dentin specimens were etched with phosphoric acid for 15, 30, or 60 seconds and then incubated with a primary monoclonal antibody anticollagen type 1, followed by a secondary antibody goat antimouse conjugated with gold particles of 15-nm diameter. High-resolution, in-lens field emission backscattered scanning electron microscope images revealed labeling of the collagen fibers in all specimens etched with phosphoric acid (Fig. 10). The signal emitted from the gold particles was evident in the peritubular dentin and on radial fibrils that suspend the odontoblastic tubule inside the tubule lumen. The positive labeling of the dentin specimens etched with phosphoric acid showed that the acid unveiled the antigenic binding sites of the collagen fibers by dissolving the mineral crystals. After 15 seconds of etching, the labeling was higher than when acid was applied for 30 seconds or 1 minute. These results provide additional evidence that a 15-second application of phosphoric acid results in a mineral dissolution of the crystals, enveloping the superficial collagen fibers without damaging the collagen ultrastructure. [28]

When the "wet-bonding" technique [18] was first considered a common procedure associated with dentin bonding, all manufacturers began recommending a moist dentin surface as the ideal substrate. The demineralized dentin matrix has been described to collapse easily when it is dried with air after being rinsed with water. [29–31] After removal of the hydroxyapatite

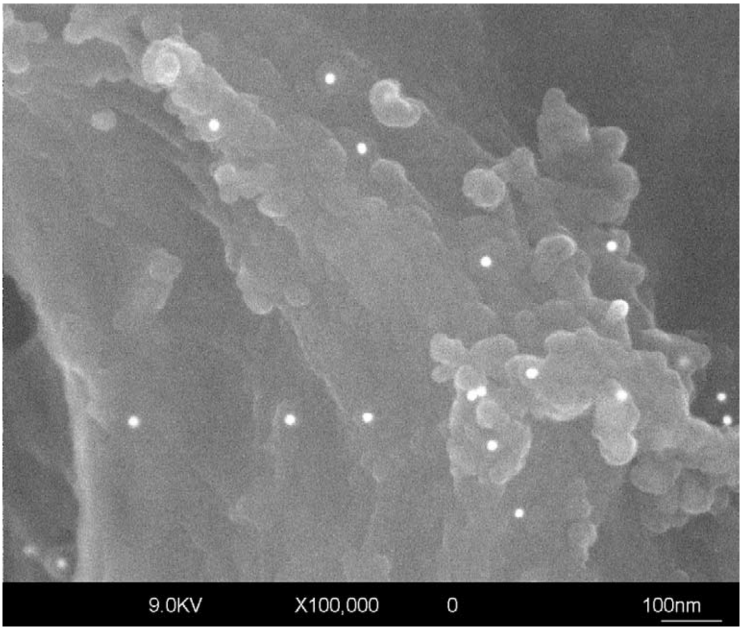

Fig. 10. Nano-gold particles bound to collagen.

crystals by acid etching in vitro, it is crucial to keep the tissue moist when rinsing off the etchant (Fig. 11) so that the collapse by air drying of the fibrillar structure of the collagen scaffold at the superficial demineralized zone is prevented. [18,31] It has been reported that the infiltration of the adhesive monomers through the nanospaces of the moist, dense collagen web enhances bond strengths in vitro. [18,31,32] Current dentin adhesives are usually dissolved in acetone or ethanol. It is thought that these organic solvents can displace water from the dentin surface and from the moist collagen network [18,32,33] to allow the monomers to intermingle with the exposed collagen fibers (Fig. 12) and form a "hybrid layer." [34]

In dental schools, air drying of etched cavities used to be taught as a method to check for an adequate etched aspect of enamel. Consequently, many clinicians still dry the cavity preparation after rinsing the etching gel to check for that frosted aspect. It is clinically impossible to dry enamel without drying dentin simultaneously. Consequently, as a result of air drying enamel, dentin is also dried, which causes dentin collagen to fall down, resulting in the closing of the pores in intertubular collagen in vitro. [31] Re-wetting dried dentin with water raises the collapsed collagen to a level compared with a wet-bonding technique and restores the bond strengths when dentin is re-wet for twice as long as the time spent with drying (Fig. 13). [35–37]

In-vitro research data focused on different degrees of moisture have not been corroborated by clinical findings. As opposed to laboratorial condi-

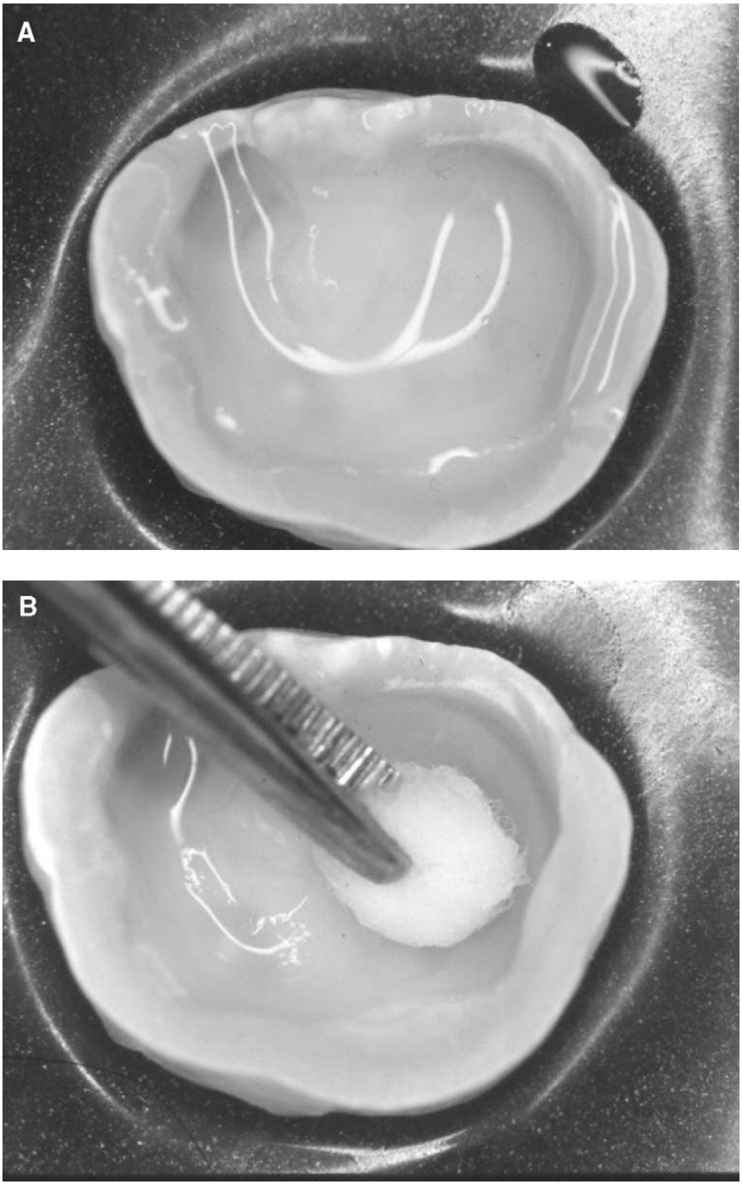

Fig. 11. (A) Overwet preparation upon rinsing off the phosphoric acid gel. (B) A damp cotton pellet is used to remove the excess of water without causing desiccation. (C) Preparation is left visibly moist and ready to be impregnated by the dentin adhesive.

tions, dentin is an intrinsically hydrated tissue in vivo, penetrated by a network of 1.0- to 2.5-μm diameter fluid-filled dentin tubules. Flow of fluid from the pulp to the dentin-enamel junction is the result of a slight but constant pulpal pressure. [38] Pulpal pressure has a magnitude of 25 to 30 mm

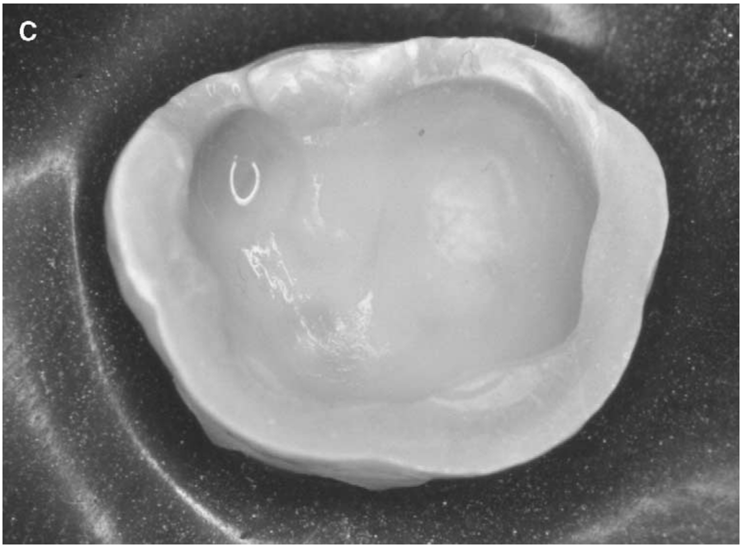

Fig. 11 (*continued*)

Hg or 34 to 40 cm H_2O. [39,40] Six-month results of a clinical trial of two dentin adhesives applied on moist versus dry dentin have recently shown that the moisture level of the substrate may not be as important clinically as it is under laboratorial conditions. [41] In this study, Single Bond (3M ESPE, St. Paul, MN), an ethanol- and water-based adhesive, and Prime & Bond NT (Dentsply Caulk, Milford, DE), an acetone-based adhesive, were applied in noncarious, nonbeveled, class V cavities either after removing the excess of water with a damp cotton pellet or after drying enamel and dentin with air for 2 to 3 seconds. Retention rates for both adhesives applied on dry dentin were 100% at 6 months, which challenges both the clinical relevance of the in-vitro findings and the experimental set-up of current laboratory experiments.

New adhesion philosophies

Recent developments in dental adhesion include "all-in-one" dental adhesive systems (see Tables 1 and 2), which fall into the family of self-etching adhesive materials. [42] The same solution serves as conditioner, primer, and adhesive. One of these "all-in-one" materials, Prompt L-Pop (3M ESPE), is a solution consisting of methacrylate phosphates, water, and a fluoride complex in a unique application unit.

The rationale behind the action of self-etching agents is the formation of a continuum between tooth surfaces and adhesive material, which is accomplished by the simultaneous demineralization and penetration of resin in enamel and dentin surfaces. Omitting the conventional phosphoric acid-

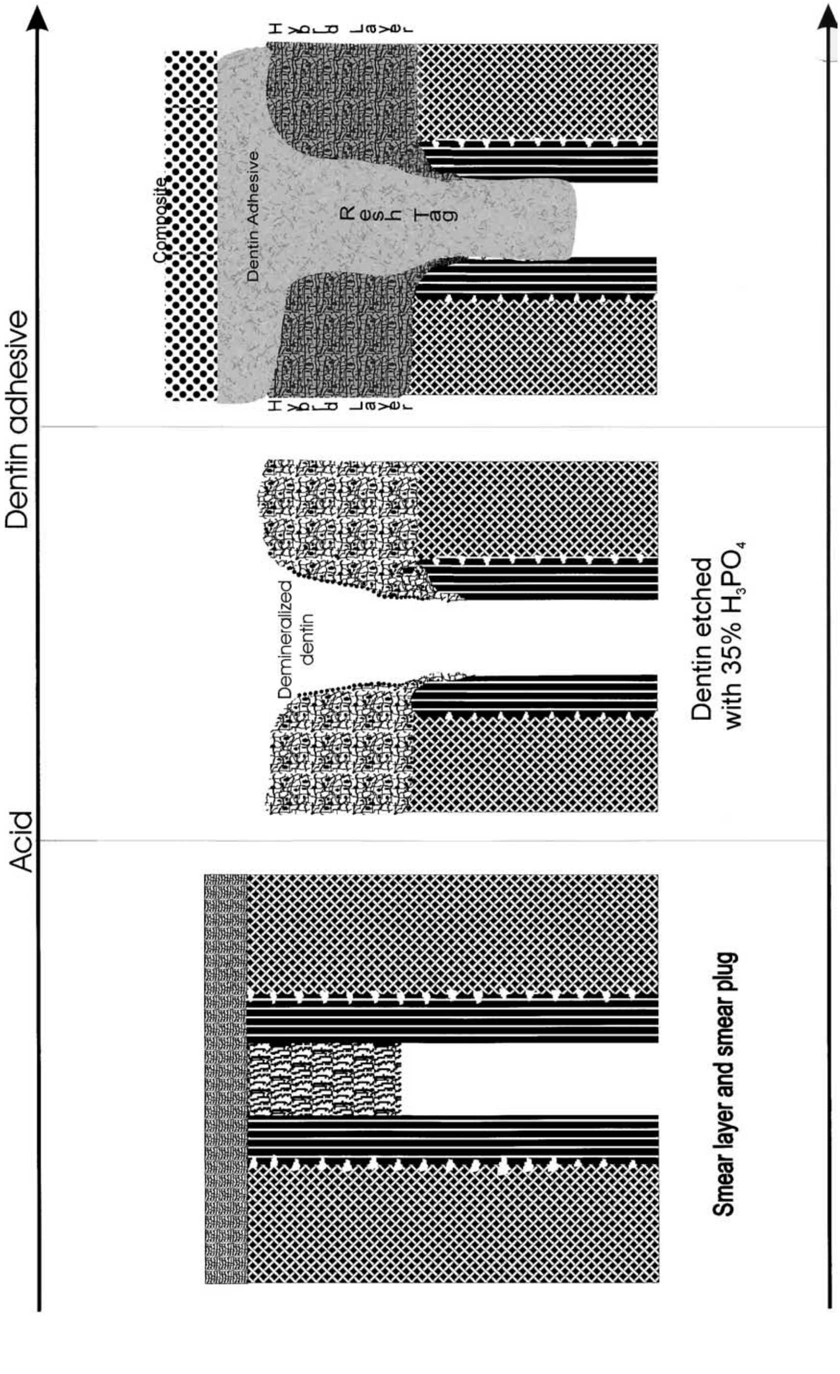

Fig. 12. Dentin hybridization.

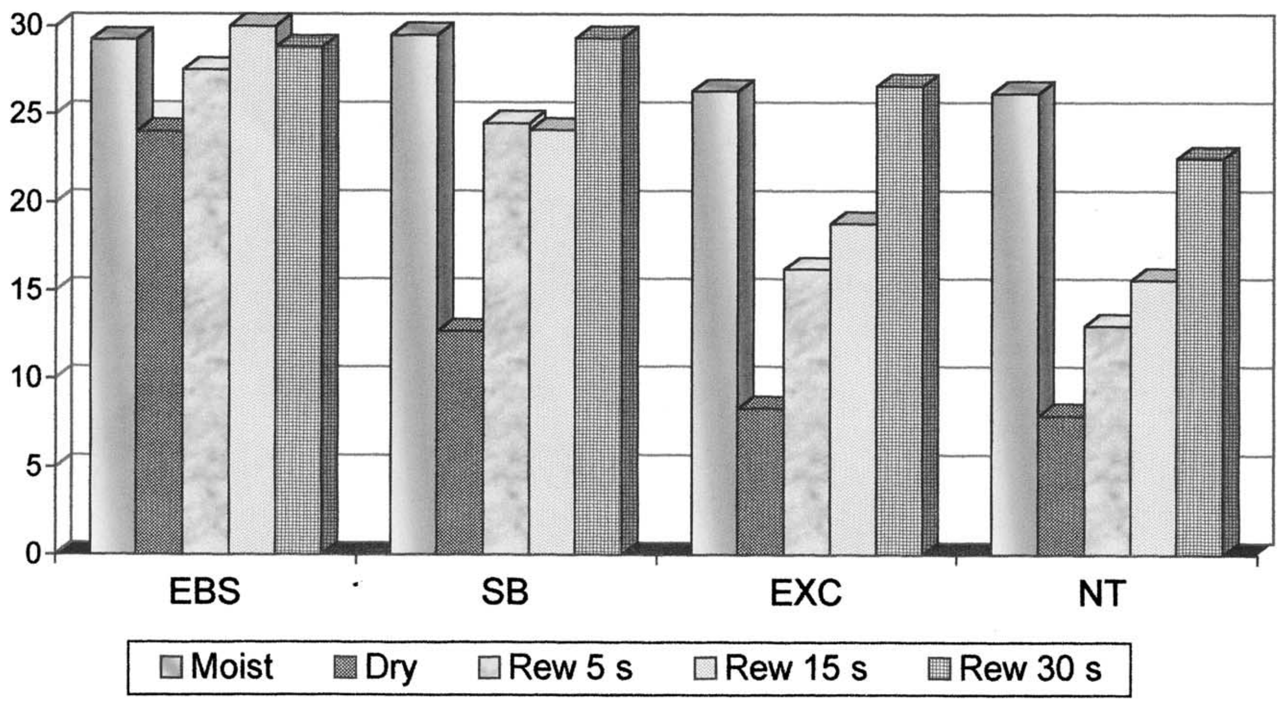

Fig. 13. Effect of re-wetting on dentin bond strengths. [36] **EBS** = EBS-Multi (water-based total-etch adhesive); **EXC** = Excite (ethanol-based total-etch adhesive); **NT** = Prime & Bond NT (acetone-based total-etch adhesive); **SB** = Single Bond (water-and-ethanol-based total-etch adhesive [3M ESPE]); **Moist** = apply adhesive on moist dentin; **Dry** = dry with air for 15 seconds prior to applying the adhesive; **Rew 5 s** = dry for 15 seconds, rewet with water for 5 seconds, apply adhesive; **Rew 15 s** = dry for 15 seconds, rewet with water for 15 seconds, apply adhesive; **Rew 30 s** = dry for 15 seconds, rewet with water for 30 seconds, apply adhesive.

etching step with self-etching materials, however, may result in the absence of the characteristic demineralization of enamel and dentin (Fig. 14), especially for unprepared enamel [43] and sclerotic dentin. [44] Under the scanning electron microscope, the etching pattern of enamel achieved by Prompt L-Pop (3M ESPE) is similar to that created by phosphoric acid when enamel is prepared. [45,46] The enamel shear bond strengths are higher, however, when Prompt L-Pop is combined with a compomer than when it is combined with a composite. [47] A viable explanation would be the low viscosity of the compomer and the fact that Prompt L-Pop is a water-based material and therefore chemically more compatible with restorative materials with enhanced hydrophilic properties. When enamel is not instrumented, Prompt L-Pop results in a poorly defined etching pattern (Fig. 15).

Etching enamel with nonrinsing conditioners of a pH higher than that of phosphoric acid remains controversial in terms of the clinical effectiveness of the conditioners and the durability of the restoration. [48] At 1 year, there is some clinical evidence of marginal defects on enamel treated with self-etching adhesive materials. [49] As a consequence of this unpredictable behavior around the enamel margins, etching with 30% to 40% phosphoric acid is still recommended clinically.

"All-in-one" dentin adhesives are now increasingly used in pediatric dentistry. The hybridization that these simplified self-etching materials create in primary dentin is consistent with and similar to the hybridization provided by total-etch dentin adhesives (Fig. 16).

Testing dentin adhesives

Clinical trials

Clinical trials are the most suitable tool to evaluate the efficacy of dentin adhesive systems; however, long-term clinical trials are difficult to perform because of the time and number of patients involved. The most crucial reason that clinical trials with dentin adhesives are not common may be that manufacturers often introduce a new version of a dentin adhesive system even before the conclusion of an ongoing study, making new materials quickly archaic. Accordingly, to predict the clinical behavior of their proprietary adhesive materials, manufacturers still largely rely on laboratorial studies.

As per the American Dental Association guidelines for provisional acceptance of dentin and enamel adhesive materials, retention rates at 6 months must be at least 95%, whereas for full acceptance, retention rates must be at least 90% after only 18 months of clinical use. [50] Several reports have been published describing the clinical behavior of total-etch multibottle adhesive systems. [51–54] Retention rates have varied from 69% to 100% up to 3 years. Regarding the clinical behavior of "one-bottle" adhesives, retention rates for OptiBond SOLO (Kerr Co., Orange, CA) and for Prime & Bond 2.1 (Dentsply) at 18 months were close to 100% for both dentin adhesives

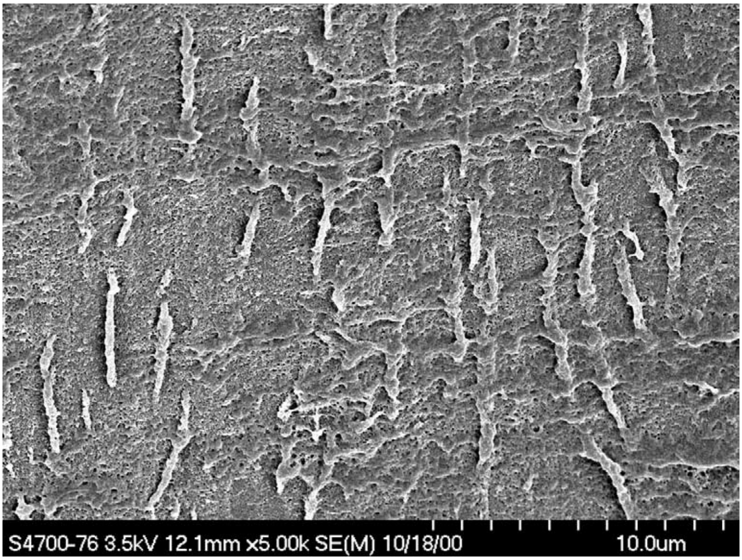

Fig. 14. Poor etching pattern (replica) of instrumented enamel as a result of the application of an experimental self-etching material.

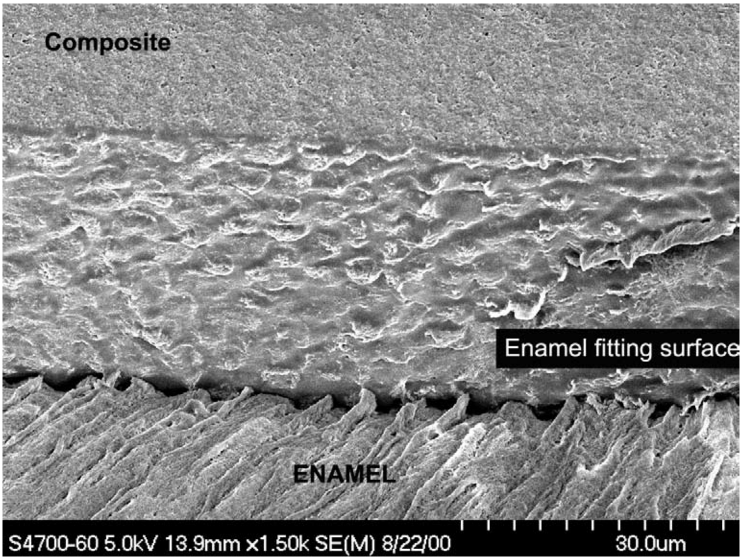

Fig. 15. Fitting surface of non-instrumented enamel upon treatment with Prompt L-Pop (3M ESPE). Specimen tilted at 45°.

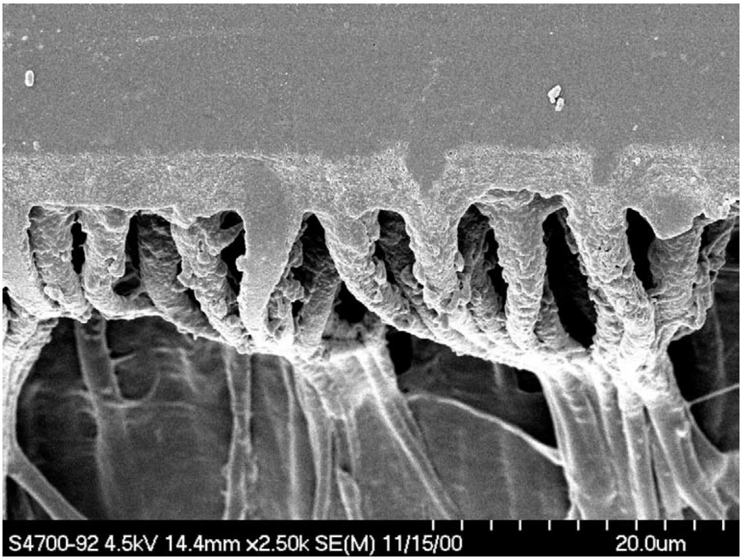

Fig. 16. Primary dentin: hybridization with Prompt L-Pop (3M ESPE). Dentin has been dissolved chemically.

[55]. For Prime & Bond NT (Dentsply), when dentin was etched with 36% phosphoric acid, retention rates at 18 months were in the range of 92%. [48] When a nonrinsing conditioner composed of maleic and itaconic acids in water (NRC, Dentsply DeTrey, Konstanz, Germany) was used instead of phosphoric acid, retention rates dropped to 71% at 18 months. [48] The same study reported that when Prime & Bond NT was used with the respective compomer without etching (as recommended for compomer restorations), retention rates at 18 months were 72%, well below the American Dental Association's threshold for full acceptance. Retention rates for One-Step (Bisco, Schaumburg, IL) were 95% at 1 year. [56] Another study with One-Step reported a 32% retention rate to nonsclerotic dentin at 3 years. [57]

More recently, clinical studies have focused on "all-in-one" dentin adhesives. Most studies with Prompt L-Pop (3M ESPE) showed an acceptable clinical performance in class I, class III, and class V composite restorations from 6 months to 1 year. [58–61] One independent class V study with Prompt L-Pop resulted in a low retention rate of 76% at 6 months. [62]

Taking into consideration that most recent total-etch adhesives result in a clinical success rate higher than 90% at 1 to 2 years when applied to class V cavities, [48,55] clinical studies should focus on parameters other than clinical retention to allow for the differentiation in efficacy among different adhesives. As per the American Dental Association guidelines, clinical studies with adhesive materials must be conducted in noncarious class V lesions. The dentin substrate in noncarious class V cavities is usually hypermineralized, which may not be as clinically relevant as if it were carious dentin. [44,63] The inclusion of carious lesions in clinical trials with dentin adhesives might increase the clinical relevance of these studies. Additionally, a "retained" restoration is always assessed as "bonded" to the cavity walls, yet retention and bonding are two different concepts. A composite restoration might be retained in a class V cavity, even without macromechanical retention features, without being totally bonded at the resin-dentin interface.

The type of composite is thought to play an important role in clinical longevity of class V restorations. Microfilled composites have a low Young's modulus, which means that they are more able to relieve stresses caused by polymerization contraction or by tooth flexure. [64,65] Materials that have a higher Young's modulus do not dissipate stresses by flow, and therefore are unable to compensate for the stresses accumulated during polymerization. These stresses may be subsequently transmitted to the interface and cause detachment of the restoration. [65] This physical concept was generally accepted when third-generation dentin adhesives were being used. [66] For total-etch dentin adhesives, clinical studies have shown that there is no significant difference in clinical behavior between low- and high-modulus restorative materials in noncarious class V restorations. [51,67] The application of phosphoric acid on dentin is now more consistent than the type of restorative material for predicting retention of class V restorations.

Laboratory tests

It has been demonstrated that testing methods using smaller surface areas generate higher bond strengths than those methods using larger surface areas. [68] The inconsistency of tests with large surface areas, such as the conventional shear bond strength testing, is probably a result of greater number of defects occurring in large surface areas than in smaller areas. [69] Besides resulting in higher bond strengths than conventional methods, the new microtensile method [70] (Fig. 17) tends to result in more adhesive failures than cohesive failures because the stress distribution is more uniform. Microtensile bond strength testing enables the investigation of interfacial bonds with reduced probability of pulling out dentin from the flat surface, as often recorded when testing in shear or conventional tensile mode. [70–72] The microtensile test produces a more reliable measurement of bond strengths than the conventional shear bond test, which undergoes unbalanced force distribution during testing. [68] With the microtensile method, it is possible to unveil differences among materials that were not observed with shear and tensile bond tests. [73] Because the microtensile method measures bond strengths in small areas, it permits several measurements from a single tooth, which facilitates the use of human teeth. [70] The use of the nontrimming technique (Fig. 18) with microtensile bond testing further allows the comparison of bond strengths between different areas of the same tooth.

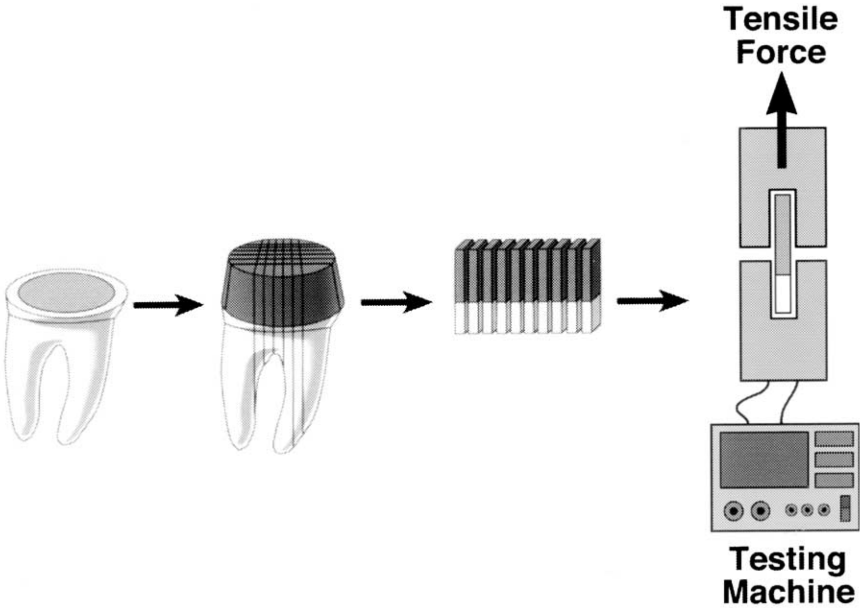

Fig. 17. The non-trimming technique for the microtensile test.

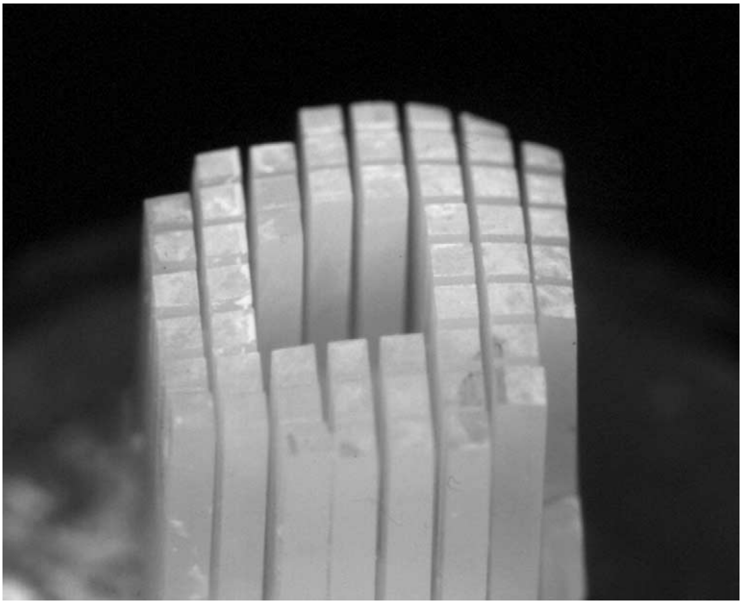

Fig. 18. The non-trimming technique allows testing of several areas of the same tooth with the microtensile test.

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