

Polymerization Shrinkage/Stress and Dentin Bond Strength of Silorane and Dimethacrylate-Based Dental Composites

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ABSTRACT: This study was designed to determine whether a new dedicated adhesive system using a silorane composite exhibits better bonding performance to human dentin than conventional dimethacrylate-based composites. The materials were used included: AdperTM Easy Bond-Z250 (AE-Z250), iBond-Venus (IB-VE), XenoIII-TPH (XE-TPH), Clearfil S3-Clearfil Majesty (S3-CM), and the Filtek silorane system (SA-FS). Polymerization volumetric shrinkage and stress development were measured using a micro-CT instrument and universal testing machine. The push out strength of the bonds produced using the corresponding self-etching adhesive systems were also measured. The volumetric shrinkage of the resin composite/adhesive combinations ranged from 1.05% (SA-FS) to 3.38% (XE-TPH) 30 min after light curing. SA-FS had the lowest volumetric shrinkage ($P < 0.05$), followed by S3-CM, EA-Z250, IB-VE,

and XE-TPH. The polymerization stress of the materials ranged from 1.54 (SA-FS) to 3.49 MPa (S3-CM). The lowest stress was also observed in SA-FS at 30 min during the stress test ($P < 0.05$). Push-out bond strength testing revealed that IB-VE had significantly lower bond strength than other combinations ($P < 0.05$). The silorane composite and dedicated adhesive system exhibited excellent characteristics of low volumetric shrinkage and stress development compared to conventional dimethacrylate-based composites. However, the silorane composite resin system possessed similar push-out bond strength as the other materials, with the exception of the Venus/iBond combination. © 2011 Wiley Periodicals, Inc. *J Appl Polym Sci* 124: 436–443, 2012

Key words: polymerization shrinkage; polymerization stress; push-out bond strength; micro-CT; silorane

INTRODUCTION

Visible light-cured composites consisting of inorganic fillers and a polydimethacrylate matrix are increasingly used as dental restorative materials because of their good esthetic and mechanical properties.^{1–3} However, the major problem that affects their performance is the intrinsic polymerization shrinkage, which is an inevitable effect of the curing process as monomer molecules, are converted into a crosslinked polymer network, exchanging van der Waals bonds for shorter covalent bonds.^{4,5} This volumetric shrinkage causes stress in confined environments such as tooth cavities.⁶ Polymerization shrinkage and the resulting shrinkage stress play an important role in influencing the forces acting on the tooth-restoration interface, because they might reduce the integrity of the restored tooth tissue and might possibly lead to

bond failure between the resin and the tooth structure. Debonding may create plastic deformation, marginal leakage, and staining,⁷ postoperative sensitivity, and increase the risk of secondary caries formation and pulpal inflammation.^{8,9}

Significant efforts have been focused on reducing the shrinkage of resin composites through improvements in chemistry and composition as well as developing methods to prevent microleakage by improving or optimizing tooth-composite bonding.¹⁰ One approach is to tailor the components of the material, such as the amount and type of matrix monomer, the filler content, the initiator level, and the addition of nonbonding microfiller particles.¹¹ Other approaches modify the application technique, such as sandwich restorations using glass ionomer, “soft starts” (controlling the reaction rate by varying the energy of the initiating lamp), incremental insertion techniques, and flexible or low-viscosity intermediate adhesives.^{12–14} Although these measurements have reduced shrinkage to a certain degree, perfect restorations with low microleakage cannot be guaranteed.

A recently introduced low-shrinkage resin-based composite (Filtek Silorane, 3M ESPE, St. Paul, MN) uses a dedicated two-step self-etch adhesive system,

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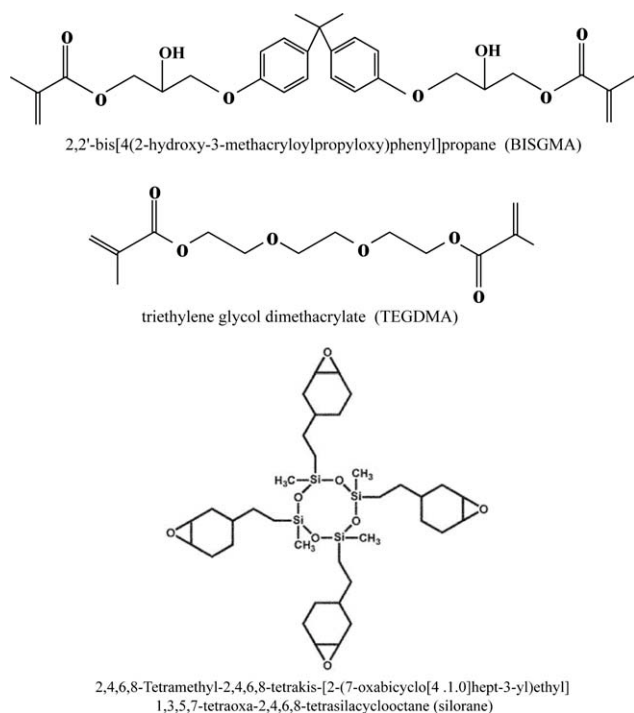


Figure 1 Chemical structure of silorane and the dimethacrylate resin monomers.

the formulation of which is claimed by the manufacturer to specifically match the chemistry of the silorane.^{15,16} Silorane is a derivative of siloxane and oxirane monomers consisting of a hydrophobic silorane backbone with oxirane rings (Fig. 1). These monomers polymerize with a cationic ring opening and exhibit less shrinkage than conventional dimethacrylate resin monomers such as Bis-GMA and TEGDMA (Fig. 1).^{17,18}

Previously, in our initial research stage, we have compared the differences of polymerization shrinkage, stress, and the degree of conversion between the silorane-based composite and conventional dimethacrylate based composites in special tooting. The results showed that silorane-based composite exhibited lower polymerization shrinkage and comparable polymerization kinetics patterns.¹⁹ However, when composite shrinkage is restricted by adhesion to the cavity walls, the compliance of the bonding substrate (which is characterized in terms of stiffness and mobility) must be considered.⁶ A layer of unfilled adhesive resin placed between the tooth and the composite restoration has been shown to absorb some of the stresses generated in the composite during polymerization and to reduce interfacial leakage.⁵ Various dentin-bonding systems have been developed to enhance the bond strength of conventional dimethacrylate-based resin composites to tooth tissues.^{20,21} Shrinkage stress is an important factor that affects the longevity of resin composite restorations, and also a direct relationship between

shrinkage stress and marginal integrity has been demonstrated.^{5,22} As the resin composites are bonded to tooth cavity walls, shrinkage stress will transfer to the bonding interface. The outcome depends on the bond strength and the mechanical properties of the structure involved. Materials under polymerization shrinkage stress combined with an advanced agent for bonding to the enamel and dentin may markedly decrease the gap formation at the bonding interface.

Previous studies have indicated that the new silorane composite system greatly reduces shrinkage^{23,24} and decreases cuspal deflection.²⁵ It is unclear whether the silorane composite with its lower shrinkage on dentin offers higher bond strength, and, to date, there have been few published studies comparing volumetric shrinkage, stress, and push-out bond strength of this adhesive system to conventional dimethacrylate-based self-etching systems. The purpose of using dentin as the substrate was to evaluate the performance of various composites (dimethacrylate vs. silorane) under simulated clinical conditions. The aim of the present study was to investigate polymerization shrinkage, stress, and bond strength to dentin of different resin composites in combination with their compatible adhesive systems and to investigate whether the new low-shrinkage silorane composite system provides higher bond strength. We hypothesized that the new silorane composite combined with the dedicated adhesive system would exhibit better bonding performance than conventional dimethacrylate-based resin composites.

MATERIALS AND METHODS

S3-CM, EA-Z250, IB-VE, and XE-TPH were used to be control groups of conventional dimethacrylate-based dental composites systems. To avoid incompatibility between the products of different manufacturers, five resin composite materials and five corresponding self-etching adhesive systems were selected as outlined in Table I. All resin composites were of the A₂ shade.

Polymerization volumetric shrinkage test

A total of 30 intact freshly extracted caries-free human molars were selected and stored in distilled water-containing 0.5% sodium azide. The occlusal enamel layers were removed to obtain a flat surface, and the teeth were randomly divided into five groups ($N = 6$). An occlusal cylindrical cavity with a depth of 2 mm and a diameter of 4 mm was prepared using a high-speed handpiece with a cylindrical medium-grit (100 μm), water-cooled diamond bur. The dentin surface of each tooth was treated

TABLE I
Materials Used in the Study

| Group | Adhesive | Composite resin | Manufacturer |
|---------|--|--|------------------------------------|
| IB-VE | iBond: UDMA, 4-META, glutaraldehyde, acetone, water, photoinitiators, and stabilizers | Venus: Bis-GMA and TEGDMA; filler: 57 vol % filler; photoinitiator | Heraeus Kulzer, Hanau, Germany |
| XE-TPH | Xeno III: Liquid A: HEMA, purified water, ethanolurethane, dimethacrylate resin, BHT, and highly dispersed SiO ₂ Liquid B: phosphoric acid modified polymethacrylate resin, PEM-F, modified Methacrylate resin, UDMA, ethyl-4-dimethylaminobenzoate, and CQ | TPH: Bis-GMA, Bis-EMA, TEGDMA; filler: 57 vol %; photoinitiator | DENTSPLY DeTrey, Konstanz, Germany |
| SA-FS | Filtek silorane adhesive: Primer: HEMA, BisGMA, water, ethanol, phosphoric acid-methacryloxy-hexyl esters, silane-treated SiO ₂ , 1,6-hexanediol dimethacrylate, copolymer of acrylic and itaconic acid, and photoinitiator Bond: substituted Dimethacrylate, silane-treated silica, TEGDMA, phosphoric acid methacryloxy-hexyl esters, 1,6-hexanediol dimethacrylate, and CQ | Filtek silorane (FS) 3,4-epoxy-cyclohexylethyl-cyclopoly-methylsiloxane, bis-3,4-epoxycyclohexyl-ethylphenylmethylsilane; filler: 57 vol %; photoinitiator | 3M ESPE, St. Paul, MN, USA |
| S3-CM | Clearfil S3: Bond: MDP, Bis-GMA, HEMA, photoinitiators, ethanol, water, and silanized colloidal silica | Clearfil Majesty posterior (CM): Bis-GMA, TEGDMA, and ArDMA; filler: 82 vol %; photoinitiator | Kuraray, Okayama, Japan |
| EB-Z250 | Adper TM easy bond: HEMA, Bis-GMA, water, phosphoric acid- methacryloxy-hexyl esters, ethanol, silane-treated silica, HDDMA, copolymer of acrylic and itaconic acid, DMAEMA, phosphine oxide, and CQ | Z250: Bis-GMA, bis-EMA, UDMA, and TEGDMA; filler: 60 vol %; photoinitiator | 3M ESPE, St. Paul, MN, USA |

Abbreviations: HEMA, 2-hydroxyethyl methacrylate; Bis-GMA, bisphenol A diglycidyl methacrylate; TEGDMA, triethylene-glycoldimethacrylate; BHT, butylated hydroxy toluene; PEM-F, pentamethacryloyloxyethylcyclohexaphosphazene monofluoride; UDMA, urethane dimethacrylate; MDP, 10-methacryloyloxydecyl dihydrogen phosphate; DMAEMA, dimethylaminoethyl methacrylate; HDDMA, 1,6-hexanediol dimethacrylate; ArDMA, aromatic dimethacrylate; 4-META, 4-methacryloxyethyltrimellitic acid; CQ, camphorquinone.

with the manufacturer-recommended self-etching adhesive system, and the cavity was filled with the corresponding restorative composite.

The specimens were examined using a high-resolution computerized X-ray microtomography apparatus (SIEMENS Inveon, Germany) to evaluate the volumetric shrinkage during the curing process. The scanning parameters were set at 14.95- μm line resolution and 800-ms exposure time using an X-ray source operated at 80 kVp and 500 μA . The tooth cavities were filled with uncured resin composite and fixed upright at marked positions in the micro-CT sample holder for scanning. The composites were then light-cured for 40 s using a source located 1 mm above the top of the composite (450 mW/cm² light intensity, QHL75, DENTSPLY, Germany). A second scan was carried out 30 min after polymerization. 3D images were reconstructed and analyzed using the manufacturer's Inveon imaging and evaluation software and Image J image analysis software. The shrinkage was calculated using the following equation: $dV = (V_0 - V_1)/V_0 \times 100\%$ (dV , volumet-

ric shrinkage; V_0 , volume before curing; V_1 , volume after curing). Internal pores were detected in all tested composites by micro-CT. The average volumetric shrinkage was calculated for the six samples in each group.

Statistical analysis was performed using one-way ANOVA and Tukey's *post hoc* test for multiple comparisons with a significance level of 0.05.

Polymerization stress measurement

The polymerization stress was determined with a universal testing machine (EZ-Test, Shimadzu, Japan) with a fixture holding the composite material on a glass plate parallel to the flat surface of the dentin.^{26–28} The occlusal enamel layers of 30 intact caries-free molar teeth were removed using a water-cooled low-speed diamond saw, leaving ~ 3 mm between the free dentin surface and the pulpal roof.²⁹ Specimens consisting of standardized cylinders of dentin ($\Phi = 6.0$ mm) were obtained using a hollow diamond drill with copious water cooling.

TABLE II
Means and SDs (in Brackets) of Polymerization Volumetric Shrinkage (%),
Stress (MPa), and Push-Out Bond Strength (MPa) for Silorane and
Dimethacrylate-Based Dental Composites

| Group | Volumetric shrinkage (%) | Stress (MPa) | Push out bond strength (MPa) |
|---------|--------------------------|--------------------------|------------------------------|
| SA-FS | 1.05 (0.09) ^a | 1.54 (0.15) ^a | 18.38 (1.70) ^a |
| EA-Z250 | 2.59 (0.20) ^b | 2.02 (0.22) ^b | 17.53 (1.21) ^a |
| S3-CM | 2.02 (0.11) ^c | 3.49 (0.34) ^c | 16.64 (1.42) ^a |
| IB-VE | 3.17 (0.25) ^d | 3.12 (0.30) ^d | 12.88 (2.28) ^b |
| XE-TPH | 3.38 (0.17) ^d | 3.04 (0.25) ^d | 17.51 (1.50) ^a |

For each property, the values in a column with the same lowercase letters indicate no significant difference ($P > 0.05$).

Waterproof abrasive paper (600-grit) was used to simulate a smear layer on the free flat surface of the specimens.

Before bonding, the surface of the glass plate where the resin composite was to be adhered was wet ground on 600-grit SiC sandpaper, sandblasted with Al₂O₃ (50 μm), and cleaned with compressed air. After water spraying and drying, the glass plate was mounted to the stationary framework of the universal testing machine. The site of the resin composite adhering to the glass was coincident with the dentin specimen mentioned earlier. The surface of the dentin was treated using one of the bonding systems described in Table I, and the specimen was immediately fixed in the steel specimen holder and connected to the crosshead of the universal testing machine. A sufficient amount of resin was applied to the dentin surface, and the cross-head was lowered toward the glass plate until the distance between the dentin surface and the glass surface was 2mm. Excess resin was removed while shaping the specimen to a cylindrical form. Throughout the measurement, the axial shrinkage of the specimens was continuously counteracted by the feedback displacement of the crosshead (controlled by the computer) to keep the specimen height constant during the test.²⁸

The composite sample was light-cured for 40 s (QHL75, Dentsply, Germany). The axial polymerization stress development was recorded continuously from the start of curing until 30 min had elapsed.

One-way ANOVA and Tukey's *post hoc* tests were used to analyze differences ($P < 0.05$) in the polymerization stress at 30 min within each group. The number of experiments was $n = 6$ for each group.

Push-out bond strength test

Twenty-five dentin disks of 2.0 mm thickness were prepared from 25 teeth perpendicular to the tooth axis using a water-cooled low-speed diamond saw.

Three standardized occlusal cavities of 2.00 mm top diameter and 1.5 mm bottom diameter were prepared into each disk using a conical-shaped diamond bur in a high speed handpiece under air-water spray coolant. The diamond bur was replaced after every fifth preparation.

The adhesive system and the composite material of each group were applied and polymerized with a light curing unit (QHL75, DENTSPLY, Germany). Specimens were stored in distilled water at 37°C for 24 h. Each dentin disk was sufficiently supported by a steel device with a central orifice. After measuring the thickness of each disk with a digital caliper, the filling material was loaded with a 1.3-mm diameter cylindrical plunger. Push-out force was applied from the bottom of the disks with the 1.5 mm diameter. Loading was performed on a universal testing machine (EZ-Test, Shimadzu, Japan) at a cross-head speed of 0.5 mm/min until bonding failure occurred. The force was recorded, and the push-out bond strength (MPa) was calculated from the values of maximum load (N) and adhesion area of resin composite filling (mm²). After testing, the failure mode was determined by inspection using a 50× stereomicroscope.

The push-out bond strength data were statistically analyzed using a one-way ANOVA and Tukey's *post hoc* test to analyze for differences ($P < 0.05$). Failure analysis data were summarized by calculating the mean percentage of interfacial failure.

RESULTS

Volumetric shrinkage

Volumetric shrinkage test results are shown in Table II. SA-FS displayed the lowest shrinkage ($P < 0.05$), followed by S3-CM, EA-Z250, IB-VE, and XE-TPH. Significant differences were observed among the dimethacrylate resin composites groups, with S3-CM and XE-TPH showing the lowest and highest volumetric values, respectively ($P < 0.05$). *Post hoc* test

exhibited no statistical difference between IB-VE and XE-TPH ($P > 0.05$).

Polymerization stress test

Figure 2 displays representative polymerization stress versus time curves and stress kinetics profiles for each material. During the 30-min measurement period, no debonding occurred between the dentin and composite. In all groups, the polymerization stress development climbed rapidly for the first minute after the start of curing and then increased at a slower rate up to 30 min. SA-FS exhibited the lowest polymerization stress after 30 min ($P < 0.05$). Among the four dimethacrylate resin composites, S3-CM stress value was significantly higher than those of EA-Z250, IB-VE, and XE-TPH ($P < 0.05$).

Push-out bond strength test

The statistical analysis of the push-out bond strength tests and the significance of differences between groups are contained in Table II. The distribution of failure modes is graphed in Figure 3.

The IB-VE system displayed significantly lowest bond strengths than other systems ($P < 0.05$). There were no significant differences between the other four groups. Adhesive failure and mixed failure were the major frequently occurring failure mode in all groups.

DISCUSSION

Volumetric shrinkage occurs as the van der Waals distance between molecules is reduced when monomers react to establish a covalent bond during polymerization. In the present study, micro-CT appara-

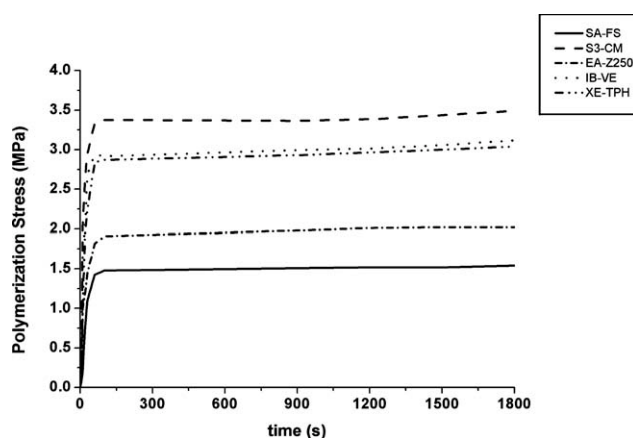


Figure 2 Development of polymerization stress. Polymerization contraction stress development in resin composite systems during a period of 30 min after light curing.

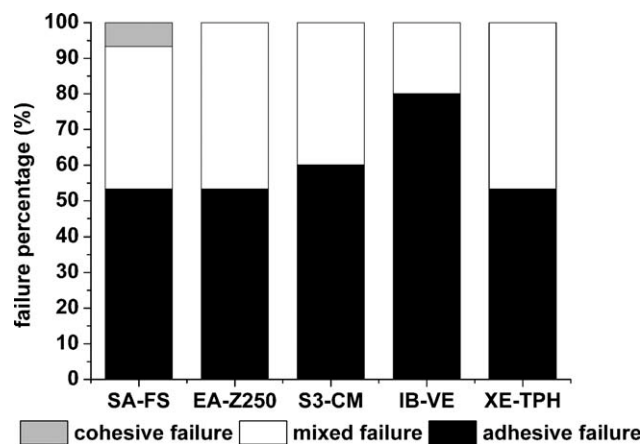


Figure 3 The distribution of failure modes. The predominant occurrence of adhesive failure and mixed failure were evident in all groups.

tus was used to visually determine the precise location, volume of resin composites, and evaluate the shrinkage of resin composites during polymerization. One-way ANOVA testing of the volumetric shrinkage data indicated that the SA-FS offered the lowest volumetric shrinkage (1.05%). This is in agreement with previous studies on silorane.^{23,24,30} The monomer system largely determines the polymerization mechanism and basic properties of resin composites. The polymerization process in the SA-FS system occurs via a cationic ring-opening reaction, which results in lower polymerization shrinkage compared to dimethacrylate-based resins that polymerize via a radical addition reaction of their double bonds. During the polymerization process, molecules must approach one another to form chemical bonds, resulting in a loss of volume. As silorane-based composites polymerize, "ring-opening" monomers connect by opening, flattening, and extending toward each other. This expansion of the cationic ring-opening of cycloaliphatic oxirane moieties balances the shortening of intermolecular distances, which reduce polymerization shrinkage.²³ When dimethacrylate-based composites are cured, the molecules are connected by actually shifting closer together, resulting in a loss of volume. Besides the chemical structure of the monomer used in the matrix resin, the properties of dental composites are also greatly influenced by the type and amount of inorganic fillers, because the overall polymerization shrinkage depends on the amount of polymer matrix present.³¹ For the dimethacrylate-based resin composites, S3-CM had significantly lower volumetric shrinkage, possibly due to a higher volume fraction of nanosized filler (82 vol %). Experiments using composites containing (BisGMA/TEGDMA) mixed with filler at various volume ratios also demonstrated a decreasing linear correlation with volume shrinkage.³²

Polymerization shrinkage stress remains a hotly debated issue, because neither determination of stress nor evaluation of its clinical impact is straightforward.³³ The stress development in polymerizing resins cured in a universal testing machine commonly exhibit a continuous increase in stress at least during the first 30 min after curing.^{34,35} The volumetric shrinkage during polymerization is dictated by the filler content and resin matrix composition and is responsible for the intrinsic contraction stress in the resin composite. According to Hooke's law, the increase in stress over a given time interval would be proportional to the increase in volumetric shrinkage by the increase in the material's elastic modulus.⁶ Furthermore, the elastic properties (i.e., *E*-modulus or tensile modulus) and the ability of the polymer to rearrange and relieve stress have been shown to influence the final stress.³⁶ In clinical cases, the polymerization stress development depends not only on the characteristics of the resin composite, but also the compliance of the bonding substrate, cavity size and geometry, and application technique.³⁷

In the measurement process, the dimethacrylate-based composites and the SA-FS bonded well with the dentin surface using the appropriate self-etching adhesive system. The polymerization stress curves of the silorane material follow the same pattern as the dimethacrylate monomers. The rising portion of the curve in the first 60 s identifies a period in which chain growth rapidly increases. The plateau occurs after a high degree of conversion and reduced stress.

SA-FS developed a very low polymerization stress compared to dimethacrylate-based composites. Also, previous literature has reported that the low-shrinkage silorane-based composites demonstrated considerable lower value in manifestation of shrinkage stress than other conventional dental composites.³⁸ On one hand, this could be attributed to differences between free radical and cationic polymerization reaction kinetics. On the other hand, it has been previously recognized that an intermediate bonding layer between the cavity walls and the composite may act as an elastic buffer. The effectiveness of the buffer depends on the thickness and distribution over the dentin surface. The higher viscosity of the silorane adhesive resin compared to the dimethacrylate-based adhesive resin could result in the formation of a high thickness layer and act as an elastic buffer that partially compensates for polymerization stress.^{39,40} The polymerization stress testing revealed uniform behavior with respect to volumetric shrinkage except in the case of S3-CM. Increased filler loading and reduced resin content result in a decrease in shrinkage. However, Condon reported a strong correlation between filler volume in commer-

cial resin composites and stress and noted that increasing filler load would result in higher stress.⁴¹ Although intrinsic strain is the only factor affecting polymerization stress outside the cavity, when composites are cured in a constrained condition other factors such as stiffness, composite viscosity, and interfacial bond strength begin to affect the polymerization stress. The S3-CM samples had the highest polymerization stress among the dimethacrylate-based composites, possibly due to the higher filler loading (82 vol % according to the manufacturer). Increasing filler content can lead to higher elastic modulus and exaggeration of the stresses generated during polymerization. In short, shrinkage and stress are engaged in a complex interplay that depends on many factors such as filler load, type of filler particles, monomer system, and bonding condition.

In our apparatus, the push-out bond strength of the Filtek silorane bond to dentin was not significantly higher than the dimethacrylate-based systems. Comparisons of the volumetric shrinkage, polymerization stress, and push-out bond strength data demonstrate that although the low shrinkage properties of the silorane approach the ideal material, it seems not necessarily to solve all adhesion problems.

Theoretically, lower stress may be beneficial to the adhesive interface and induce higher bond strength for SA-FS. However, SA-FS showed a similar bonding performance with other groups. The silorane adhesive system is composed of a hydrophilic self-etch primer and a hydrophobic viscous bond coating resin. The primer and the bond are distinguishable as two distinct layers, because they are cured separately; the actual bond to the tooth surface is realized by the primer only. The separately light-cured primer can actually be regarded as a one-step "ultra mild" self-etch adhesive with a pH of 2.7. Previous study using TEM revealed a thin interaction zone, most likely representing a combination of resin-impregnation of the smear layer and actual dentin hybridization.^{42,43} As the self-etch adhesives only interact superficially with the smear layer-covered dentin, the bond strength may be influenced by the weaker properties of the interaction zone. Also, the separate application of the high hydrophobic bond may cause uneven layer thickness and phase separation from the silorane hydrophilic primer affecting bond strength.⁴⁴

In our study, the push-out bond strength of restorative composite in a higher C-factor cavity was assessed, with development of the polymerization shrinkage stress directly on the bonding interface.⁴⁵ Among the dimethacrylate-based resin composite groups, the IB-VE had the lowest bond strength with a higher percentage of adhesive failure, failing to produce reliable adhesion to dentin. For the lower

bond strength of IB-VE, one reason is probably related to a generation of higher stress, leading to disruption of bonding to the cavity walls. On the other hand, the poor performance of IB-VE may be caused by hydrolysis of the acidic monomer 4-META and the hydrophilic monomer HEMA in the presence of water within the adhesive. These monomers are readily hydrolyzed by water upon storage at elevated temperatures or with prolonged storage.⁴⁶ Also, 4-META does not have a high bonding affinity for hydroxyapatite.^{47,48} Other factors may play a role in the weak bonding performance of IB-VE. iBond is acetone-containing adhesive. Porosities occurred at the bonding interface due to water accumulation caused by an osmotic gradient or by monomer/solvent phase separation upon evaporation of the acetone.⁴⁹

In summary, the lower bond strength and more adhesive failures were observed for IB-VE, probably due to the higher stress aforementioned generation in the bonding interface, in association with the poor bonding between adhesive and dentin. This could have triggered an initial debonding. In the clinical situation, this could result in poor marginal and internal adaptation of restorations.⁵⁰

The experimental instruments used to measure polymerization volumetric shrinkage, stress, and push-out bond strength provided important information about the differences between silorane composites and conventional dimethacrylate-based composite systems. Future experiments need focus on the long-term performance of the low-shrinkage silorane composite system under similar clinical conditions.

CONCLUSIONS

Silorane composite materials with a dedicated adhesive system display the ideal characteristics of low polymerization shrinkage and stress development when applied to dentin. The silorane composite exhibited similar push-out bond strength to the dimethacrylate-based materials after 24-h water storage.

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