

# The influence of different placement techniques on the microtensile bond strength of low-shrink silorane composite bonded to Class I cavities

J.S. Almeida e Silva, DDS, MSc ▪ Juliana Nunes Rolla, DDS, MSc ▪ Luiz Narciso Baratieri, DDS, MSc, PhD  
Sylvio Monteiro Jr., DDS, MSc, MSD, PhD

The aim of this *in vitro* study was to evaluate the microtensile bond strength ( $\mu$ TBS) of a low-shrink silorane-based composite (Filtek Silorane) and a methacrylate-based composite (Filtek Z250) to the bottom dentin of a Class I cavity using different placement techniques. Twelve third molars were used. Standard, box-type Class I cavities (6.0 x 4.0 x 2.5 mm) were prepared at the occlusal crown center, with the pulpal floor ending approximately at the midcoronal dentin. The teeth were then randomly divided into four groups, according to each placement technique: ZI—Filtek Z250 placed incrementally; ZB—Filtek Z250 placed in bulk; SI—Filtek Silorane placed incrementally; and SB—Filtek Silorane placed in bulk. Each restored third molar was subjected to microtensile bond testing after 24 hours of storage in distilled water at 37°C. After storage, each molar was

longitudinally sectioned in both axes to obtain rectangular sticks with an approximate 0.49 mm<sup>2</sup> cross-sectional area. Data were analyzed by one-way ANOVA followed by a Tukey *post hoc* test ( $P \leq 0.05$ ). After debonding, the failure modes were analyzed using a stereomicroscope.

The ZI group (72.6 MPa) showed the highest  $\mu$ TBS, followed by the ZB group (60.2 MPa), while the SI (34.4 MPa) and SB (42.6 MPa) groups demonstrated statistically significant lower bond strengths. The type of placement technique did not influence the  $\mu$ TBS of silorane-based composites to the bottom dentin of Class I cavities. The methacrylate-based composite showed superior performance, regardless of the placement technique.

Received: August 9, 2010

Accepted: November 9, 2010

The use of methacrylate-based composite resin restorative materials has been widely accepted in dental practice.<sup>1,2</sup> However, improvements aiming to reduce the inherent polymerization shrinkage of the current materials are still necessary.<sup>3,4</sup> Problems associated with polymerization shrinkage are common: imperfect marginal sealing (which can result in secondary caries), marginal staining, and postoperative sensitivity.<sup>5</sup> Polymerization shrinkage also can lead to cuspal displacement and even to cracks in healthy tooth structure.<sup>6</sup>

To minimize stress from polymerization shrinkage, efforts have been directed toward improving placement techniques, curing methods, and composite formulation.<sup>4</sup> For

methacrylate-based composites, the incremental layering technique, based on polymerizing composite layers less than 2.0 mm thick, has been considered the best way to minimize the effects of the polymerization shrinkage.<sup>7,8</sup> As for the composite formulation, many attempts have been made over the past few years to change the composite nature.<sup>9-11</sup>

A novel oxirane-based composite, called *silorane*, has been synthesized from the reaction of oxiranes and siloxane molecules. It is based on using ring-opening polymerization of the silorane molecules instead of free radical polymerization of methacrylate monomers. The ring-opening polymerization of a silorane molecule is a cationic polymerization reaction in which

no oxygen inhibition layer exists on the composite surface. It has been postulated that this new composite provides increased hydrophobicity, improved biocompatibility compared to methacrylate-based composites, and decreased polymerization shrinkage (less than 1%, whereas most of methacrylate-based composites present 2–5% volumetric shrinkage).<sup>12-16</sup> The incremental placement technique minimizes the stress from polymerization shrinkage of methacrylate-based composites.<sup>4,7,8</sup> On the other hand, manufacturers claim that there is no need for the incremental placement technique when the low-shrink Filtek Silorane composite (3M ESPE) is used; therefore, bulk placement is recommended.<sup>2</sup>

Table 1. Materials used in this study.

Materials	Material composition
Scotchbond Multi-Purpose	<i>Etchant:</i> 35% H <sub>3</sub> PO <sub>4</sub> ; <i>Primer:</i> copolymer of polyalkenoic acid, HEMA, water; <i>Resin:</i> HEMA, bis-GMA
Silorane System Adhesive Bond	TEGDMA, phosphoric acid methacryloxyhexylesters, 1,6-hexanediol dimethacrylate bis-GMA, UDMA, bis-EMA
Filtek Z250	Bis-GMA, UDMA, bis-EMA
Filtek Silorane	1,3,5,7-Tetrakis(ethyl cyclohexane epoxy)-1,3,5,7-tetramethyl cyclotetrasiloxanemethyl-bis[2-(7-oxabicyclo[4.1.0]hept-3-yl)ethyl]phenyl

Bis-GMA = bisphenol A-glycidyl dimethacrylate; TEGDMA = triethylene glycol dimethacrylate; UDMA = urethane dimethacrylate; bis-EMA = bisphenol A polyethylene glycol diether dimethacrylate; HEMA = 2-hydroxyethylmethacrylate.

Table 2. Adhesives used in this study and their respective application techniques.

Adhesive	Application technique
Scotchbond Multi-Purpose	35% H <sub>3</sub> PO <sub>4</sub> acid etch (30 seconds for enamel and 15 seconds for dentin), rinse (30 seconds), air dry, primer (30 seconds), air dry, adhesive, photocure (20 seconds).
Silorane System Adhesive Bond	Primer (15 seconds), air dry, photocure (10 seconds), adhesive, air dry, photocure (10 seconds).

The aim of this current study was to evaluate the influence of different placement techniques on the microtensile bond strength ( $\mu$ TBS) of low-shrink Filtek Silorane composite bonded to the bottom dentin of Class I cavities.

**Materials and methods**

The materials used in the present study are listed in Table 1. Twelve human third molars were stored in distilled water at 4°C, with the storage medium renewed weekly, and used within three months of extraction.

Standard, box-type, Class I cavities (6.0 x 4.0 x 2.5 mm) were prepared at the occlusal crown center with the pulpal floor ending approximately at the midcoronal dentin,

using a high-speed handpiece with cylindrical high, medium, and fine grit diamond burs (KG Sorensen), respectively. The diamond bur was positioned perpendicularly to the long axis of the tooth to create a 2.5 mm deep cavity. Such cavity depth standardization was achieved by leveling the upper portion of the diamond bur (4.0 mm long) with the tooth marginal ridges. The prepared teeth were then randomly assigned to one of four experimental groups (three molars per group): SB—Filtek Silorane placed in bulk; SI—Filtek Silorane placed incrementally; ZI—Filtek Z250 placed incrementally; and ZB—Filtek Z250 placed in bulk. All Filtek Silorane layers were less than 2.5 mm thick, and all materials were used according

to the manufacturer’s instructions and subjected to bonding treatments outlined in Table 2.

**Placement technique**

After the bonding treatment, the placement technique applied for each experimental group was performed as described below.

SB—Filtek Silorane shade A3 composite was placed in bulk with <2.5 mm thick increments and photocured for 40 seconds using a halogen photocuring device (Elipar 2500 curing light, 3M ESPE) at 400mW/cm<sup>2</sup>.

SI—Filtek Silorane shade A3 composite was placed in five increments (<2.0 mm thick) without linking the opposing cavity internal walls. Each increment was photocured for 40 seconds using a halogen photocuring device (Elipar 2500 curing light) at 400mW/cm<sup>2</sup>.

ZI—Filtek Z250 shade A3 composite was placed in five increments (<2.0 mm thick) without linking the opposing cavity internal walls. Each increment was photocured for 40 seconds using a halogen photocuring device (Elipar 2500 curing light) at 400mW/cm<sup>2</sup>.

ZB—Filtek Z250 shade A3 composite was placed in bulk with <2.5 mm thick increment and photocured for 40 seconds using a halogen photocuring device (Elipar 2500 curing light) at 400mW/cm<sup>2</sup>.

**Microtensile bond strength testing**

Each restored molar was subjected to microtensile bond testing after storage in distilled water at 37°C for 24 hours. After storage, each molar was longitudinally sectioned along both axes to obtain rectangular sticks with a cross-sectional area of approximately 0.49 mm<sup>2</sup>. The sticks then were fixed to a Geraldelli jig using cyanocrilate glue applied to

both extremities of each stick.<sup>17</sup> The sticks were stressed in a universal testing machine (Instron 4444, Instron Corp.) at a crosshead speed of 0.5 mm/min until failure. The  $\mu$ TBS was expressed by the following equation: MPa = N/mm<sup>2</sup>.

The failure mode of each stick was analyzed under 40x magnification microscopy (Olympus America) after debonding. The failure mode was determined using an adaptation of the Hashimoto classification system.<sup>18</sup> The failures were classified as adhesive (type A), resin cohesive (type B), dentin cohesive (type C), or mixed (adhesive failure with some dentin or resin cohesive involvement) (type D). The statistical analyses were conducted using ANOVA ( $P \leq 0.05$ ) and a *post-hoc* Tukey test.

## Results

### Microtensile bond strength

The overall bond strength values (MPa) and standard deviations (SDs) for the experimental groups are presented in Table 3. The ZI group demonstrated the highest  $\mu$ TBS mean value, followed by the ZB group, while the SI and SB groups indicated statistically lower  $\mu$ TBS mean values. The SI group exhibited the lowest  $\mu$ TBS mean value among all groups. One-way ANOVA and a *post-hoc* Tukey test revealed no significant correlation between placement technique and  $\mu$ TBS; however, significant differences were exhibited regarding the type of restorative material used ( $P \leq 0.05$ ).

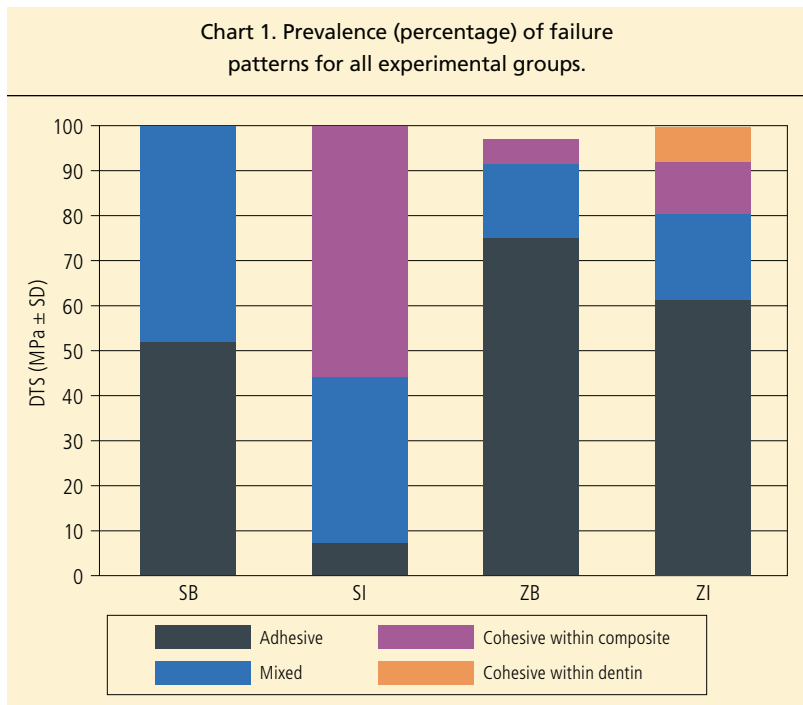
### Failure mode analysis

Chart 1 shows the proportional prevalence (percentage) of the failure patterns for all experimental groups. For the SB group, adhesive and mixed failures occurred in similar proportions. However, cohesive failure within composite was the predominant pattern for the SI group.

**Table 3.  $\mu$ TBS for each group to Class I cavity bottom dentin.**

Group	Placement technique	MPa (SD)
SB	Bulk	42.9 (17.6) <sup>a</sup>
SI	Incremental	34.4 (17.6) <sup>a</sup>
ZB	Bulk	60.2 (24.3) <sup>b</sup>
ZI	Incremental	72.6 (27.0) <sup>b</sup>

Note: Different superscript letters indicate significantly different results.



Adhesive failure was the common and predominant pattern for both the ZB and ZI groups, with a greater prevalence in the former.

## Discussion

The polymerization of a composite material is accompanied by a volume reduction that produces contraction stress where the composite contacts the tooth. These stresses, in turn, can affect the tooth-composite interface.<sup>19</sup> Presumably, a silorane composite stands as an alternative to

overcome the polymerization shrinkage stress inherent in methacrylate-based composites, and they are indicated for posterior restorations due to their less than 1% polymerization shrinkage. Therefore, the manufacturer's instructions recommend that silorane composites be placed in bulk, because there is no need for a special placement technique to minimize the polymerization shrinkage stress.

Preliminary studies of Class I, box-type cavities have shown that

there is a correlation between polymerization shrinkage and bond strength.<sup>19,20</sup> This *in vitro* study was designed to test the influence of placement technique on a silorane-based composite in a clinical “worst case scenario”—Class I cavities—to provide a high C-factor.<sup>19,20</sup> Consequently, the clinically relevant effect of polymerization stress could be assessed.

Bond strength values decrease as the cavity's C-factor increases.<sup>19,20</sup> This finding is commonly attributed to the polymerization shrinkage of composite resins; these resins transfer stress to the tooth/restoration interface during setting. However, since silorane is a low-shrink composite, it is unlikely that shrinkage stress is the cause of low bond strength values.

However, even though the low-shrinkage properties of silorane are desirable, they do not solve all of the adhesion problems.<sup>20</sup> In the current study, the  $\mu$ TBS values of a silorane-based composite bonded to Class I, cavity-bottom dentin were significantly lower than the values achieved with a methacrylate-based composite, regardless of the placement technique applied, showing that the inherent low-shrinkage property of silorane is not the ultimate factor that reduces bond strength and that the silorane system adhesive was not as effective as the traditional, methacrylate-based, three-step, etch-and-rinse adhesive.

To provide an appropriate bond between silorane composite and tooth hard tissues, a dedicated adhesive was developed by the manufacturer. The silorane system adhesive is composed of a self-etch primer and an adhesive bonding agent. The silorane primer contains hydrophilic and etching monomers that bond to hydrated dentin, while the silorane bonding agent contains

hydrophobic bifunctional monomers that match the hydrophobic silorane resin. Both must be cured separately and should be considered as one-step systems and compared with one-step systems.<sup>21</sup>

According to Duarte *et al*, the application of silorane primer on dentin produces intense intertubular decalcification, resulting in an exposed collagen network, while dentinal tubules remain blocked out by smear plugs.<sup>22,23</sup> After the application and polymerization of the silorane bond coating resin, a 1.9  $\mu$ m hybrid layer with few resin tags is observed.<sup>21,23</sup> In fact, it has been demonstrated that the hybrid layer thickness created with the silorane adhesive is thinner than that of etch-and-rinse adhesives and equivalent to that of one-step, self-etch adhesives.<sup>21</sup> The results of the present study demonstrate that a silorane-based composite could not achieve  $\mu$ TBS values as high as those for a three-step, etch-and-rinse adhesive associated with a methacrylate-based composite. This finding is logical, considering that one-step, self-etch adhesives generally perform very poorly with respect to immediate bond strength and short-term bonding effectiveness.<sup>24,25</sup>

Santini and Miletic conducted a study using 2D, confocal, micro-Raman spectroscopy.<sup>21</sup> According to their results, both the silorane primer and bond showed distinctive spectra, indicating separately cured layers of primer and bond. Although the bond was placed on the cured primer surface prior to being cured itself, Raman spectra indicated an intervening zone of approximately 1  $\mu$ m of mixed spectral intensities associated with both the primer and bond; this can be attributed to an oxygen inhibition layer remaining at the cured primer surface. This intervening zone could have acted as a weak link in the silorane bonding

system, thus causing lower  $\mu$ TBS values. With these results in mind, further research on more accurate fractography methods is necessary to assess whether failure occurs within the silorane adhesive system.

Although there was no statistical difference in the present study, the incremental technique associated with the traditional, three-step, etch-and-rinse adhesive and methacrylate-based composite achieved the highest  $\mu$ TBS value, demonstrating the effectiveness of this procedure on minimizing the effects of polymerization shrinkage.

As for the silorane-based composite, the SB group achieved higher bond strength values than the SI group. This indicates that the silorane-based composite should be placed in bulk, as recommended by the manufacturer, because bonding between successive layers depends on the reactivity of the material, since silorane composite systems are hydrophobic and no oxygen inhibition layer is present. Indeed, in the present study, the SI group failure mode analysis showed a high predominance of cohesive failures within the composite and the lowest  $\mu$ TBS values among all tested groups.

Another important finding in the present study is that chemical reactivity between successive layers decays over time, as does the bond strength between them.<sup>2</sup> As a result, the placement technique of the SI group did not take more than 20 seconds from placement until curing of the subsequent increment.

Although the bond strength of the silorane bonding agent was significantly lower than that of the methacrylate agent, this does not necessarily mean that the silorane system will not succeed clinically, because it might not require a very strong adhesive interface,

since the silorane-based interface is not exposed to the same degree of polymerization shrinkage stress as a methacrylate-based interface. Furthermore, evaluations regarding aspects such as marginal sealing, cuspal displacement, and *in vivo* longevity also are important in determining whether a composite is clinically effective.

## Conclusion

The type of placement technique did not influence the  $\mu$ TBS of a silorane-based composite to Class I, cavity-bottom dentin. The methacrylate-based composite demonstrated superior performance, regardless of placement technique.

## Author information

Drs. Silva and Rolla are postgraduate students, Operative Dentistry Division, Federal University of Santa Catarina, Florianopolis, Brazil, where Dr. Baratieri is a professor and Chair and Dr. Monteiro is a professor.

## References

- Leinfelder KF. New developments in resin restorative systems. *J Am Dent Assoc* 1997;128(5):573-581.
- Tezvergil-Mutluay A, Lassila LV, Vallittu PK. Incremental layers bonding of silorane composite: The initial bonding properties. *J Dent* 2008;36(7):560-563.
- Pearson JD, Bouschlicher MR, Boyer DB. Polymerization shrinkage forces of condensable composites. *J Dent Res* 1999;789(special issue):448.
- Deliperi S, Bardwell DN. An alternative method to reduce polymerization shrinkage in direct

- posterior composite restorations. *J Am Dent Assoc* 2002;133(10):1387-1398.
- Palin WM, Fleming, Nathwani H, Trevor B, Randall Ros C. *In vitro* cuspal deflection and microleakage of maxillary premolars restored with novel low-shrink dental composites. *Dent Mater* 2005;21(4):324-335.
  - Weinmann W, Thalacker C, Guggenberger R. Siloranes in dental composites. *Dent Mater* 2005;21(1):68-74.
  - Davidson CL. Resisting the curing contraction with adhesive composites. *J Prosthet Dent* 1986;55(4):446-447.
  - Lutz F, Krejci I, Barabakow F. Quality and durability of marginal adaptation in bonded composite restorations. *Dent Mater* 1991;7(2):107-113.
  - Rawls HR, Wellinghoff VT, Norling BK, Leamon SH, Swynnerton NF, Wellinghoff ST. Low shrinkage resins from liquid crystal diacrylate monomers. *Polymer Preprints Am Chem Soc* 1997;38:167-168.
  - Moszner N, Volkel T, Fischer U, Rheinberger V. Polymerization of cyclic monomers, part 8. Synthesis and radical polymerization of hybrid 2-vinylcyclopropanes. *Macromol Rapid Comm* 1999;20:33-35.
  - Smith RE, Pinzino CS, Chappelow CC, Holder AJ, Kostoryz EL, Guthrie JR, Miller M, Yourtee DM, Eick JD. Photopolymerization of an expanding monomer with an aromatic dioxirane. *J Appl Polymer Sci* 2004;92(1):62-71.
  - Guggenberger R, Weinmann W. Exploring beyond methacrylates. *Am J Dent* 2000;13(Special No):82D-84D.
  - Patent No. WO 98/22521. ESPE Dental AG, Seefeld, Germany, May 28, 1998.
  - Weinmann W, Luchterhandt T, Guggenberger R, Stippschild A, Then S, Dede K. Comparative testing of volumetric shrinkage and sealing of silorane and methacrylate filling materials. *J Dent Res* 2002;81(Special issue A), abstract No. 3382.
  - Feilzer AJ, De Gee AJ, Davidson CL. Setting stress in composite resin in relation to configuration of the restoration. *J Dent Res* 1987;66(11):1639-1639.
  - Eick JD, Smith RE, Pinzino CS, Kostoryz EL. Stability of silorane dental monomers in aqueous systems. *J Dent* 2006;34(6):405-410.
  - Arcari GM, Araújo E, Baratieri LN, Lopes GC. Microtensile bond strength of a nanofilled composite resin to human dentin after nonvital tooth bleaching. *J Adhes Dent* 2007;9(3):333-340.

- Hashimoto M, Ohno H, Kaga M, Sano H, Tay FR, Oguchi H, Araki Y, Kubota M. Over-etching effects on micro-tensile bond strength and failure patterns for two dentin bonding systems. *J Dent* 2002;30(2-3):99-105.
- Ilie N, Kunzelmann KH, Hickel R. Evaluation of micro-tensile bond strengths of composite materials in comparison to their polymerization shrinkage. *Dent Mater* 2006;22(7):593-601.
- Van Ende A, De Munck J, Mine A, Lambrechts P, Van Meerbeek B. Does a low-shrinking composite induce less stress at the adhesive interface? *Dent Mater* 2010;26(3):215-222. Epub 2009 Nov 11. doi:10.1016/j.dental.2009.10.003.
- Santini A, Miletic V. Comparison of the hybrid layer formed by silorane adhesive, one-step self-etch and etch and rinse systems using confocal micro-Raman spectroscopy and SEM. *J Dent* 2008;36(9):683-691.
- Duarte S Jr, Phark JH, Varjao FM, Sadan A. Nanoleakage, ultramorphological characteristics, and microtensile bond strengths of a new low-shrinkage composite to dentin after artificial aging. *Dent Mater* 2009;25(5):589-600.
- Duarte S Jr, Botta AC, Phark JH, Sadan A. Selected mechanical and physical properties and clinical application of a new low-shrinkage composite restoration. *Quintessence Int* 2009;40(8):631-638.
- Peumans M, Kanumilli P, De Munck J, Van Landuyt K, Lambrechts P, Van Meerbeek B. Clinical effectiveness of contemporary adhesives: A systematic review of current clinical trials. *Dent Mater* 2005;21(9):864-881.
- De Munck J, Van Landuyt K, Peumans M, Poitevin A, Lambrechts P, Braem M, Van Meerbeek B. A critical review of the durability of adhesion to tooth tissue: Methods and results. *J Dent Res* 2005;84(2):118-132.

## Manufacturers

Instron Corp., Canton, MA  
800.564.8378, www.instron.com

KG Sorensen, Barueri, SP, Brazil  
55.11.4197.1700, www.kgsorensen.com.br

Olympus America, Center Valley, PA  
800.446.5967, www.olympusamerica.com

3M ESPE, St. Paul, MN  
800.634.2249, solutions.3m.com