This study evaluated the shear bond strength of a resin-modified glass ionomer (RMGI) restorative material to a new silorane-based composite and a methacrylate-based composite in a sandwich technique with various combinations of surface treatments and bonding agents. Two composites, 2 bonding agents, and 4 surface preparations were used to create 16 groups with 10 specimens each. After 24 hours storage at 37°C in 100% humidity, the specimens were tested for shear bond strength; means and standard deviations were determined per group. Surface modifications did not affect the shear bond strength of the silorane or methacrylate composites to the RMGI. The new silorane composite had significantly lower bond strength to the RMGI compared to the methacrylate composite. The new silorane system adhesive agent had significantly higher bond strength to the RMGI compared to the methacrylate adhesive agent. The greatest bond strengths to the RMGI were produced when using the silorane system adhesive agent with the methacrylate composite.

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In 1972, Wilson & Kent introduced the first glass-ionomer (GI) cement to the dental market. The original GIs became well-known for their fluoride-releasing property, biocompatibility, and ability to chemically bond to hydroxyapatite, but their overall strength, wear resistance, esthetics, and multiple other physical properties were not adequate for use in many stress-bearing regions of the mouth.

The traditional methacrylate-based dental composites were first developed in the mid-1960s as a replacement for silicate cements and unfilled resins. Since then, they have greatly improved in properties and handling characteristics, such that now many providers consider this type of composite as a primary restorative material. At the outset, some physical properties were lacking, such as color stability and wear resistance. Over the years, these properties have improved greatly with changes made to the initiator, introduction of microfiller particles, and hybridization of manufacturing processes.

A few properties continue to hinder the methacrylate-based composite resins; foremost among them is polymerization shrinkage. The average methacrylate-based composite resin restorative material shrinks approximately 3%. Polymerization shrinkage has been cited to cause stress within the tooth as opposing walls are pulled toward each other. These stresses generated by the shrinkage can overcome the weaker bond to dentinal surfaces versus the stronger bond to enamel, thereby reducing the contraction stresses within the tooth by causing a gap at the dentin/restoration interface. This shrinkage and the resultant gap formation at the dentin interface has been thought to be a major cause for marginal microleakage and the resultant failure of composite restorations due to secondary caries, though that has yet to be definitively proven. Enamel-composite margins are typically free from this defect due to the much greater bond strength seen between composite and enamel than to dentin. This could in part explain the results gathered by Mjör, which showed that the vast majority of recurrent caries occur at the gingival margins, and suggests the need for a better bond at the dentin/restoration interface.

In 1985, McLean et al tried to address these shortcomings when they proposed a technique that attempted to combine the best of 2 worlds. It became known as the sandwich technique. Today it can be further specified as either open or closed. Both techniques utilize the dentinal bond that GIs can achieve by placing a layer of that material at the gingival margin of the preparation. The remainder of the preparation is then restored with a composite resin to provide superior overall strength, wear resistance, enamel bond strength, color matching, and other physical characteristics. The conventional GI had to be etched or roughened to create a mechanical bond to the composite, since a chemical bond between the 2 did not exist. The difference between the open and closed sandwich techniques is simply based on the extent of the GI placement.

In the late 1980s, resin was added to the GIs, and this hybrid genre was given the name of resin-modified glass ionomers (RMGI). The addition of resin improved many of the features of GIs. The resin allowed the material to be light-activated, with a command set. The strength, wear resistance, and enamel bond did not approach that of a traditional resin composite, but was a definite improvement over the conventional GIs. It also greatly improved the bond strength to other resin composites, solving the initial drawback of the sandwich techniques. With the advent of RMGI, the sandwich technique became less technique-sensitive and had a better bond between the RMGI and resin composite.

Recently, a unique composite, Filtek LS (LS), was developed by 3M ESPE. Instead of the traditional methacrylate-derived monomer, LS utilizes a silorane monomer ring. Many recent and ongoing studies of this new material have shown it to be similar in many ways to the methacrylate-based composites. It demonstrates “relatively higher flexural strength/modulus, fracture toughness,” and wear resistance. It’s arguable whether or not the bond strength to tooth structure is as good as the methacrylate-based composites. However, LS marginal integrity and microleakage is similar to that of methacrylate-based composites, hinting that a sandwich technique may still be advisable.

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The distinct advantage that LS has over methacrylate-based composites is its reduced polymerization shrinkage. The ring shape of the silorane monomer counteracts the shrinkage by expanding the ring into a linear dimension during the polymerization reaction. The expansion of the ring before polymerization has been shown to decrease the polymerization shrinkage to an average of 1%-1.5%). The decrease in shrinkage could be advantageous by reducing stresses on the tooth, however the currently available studies disagree on this point. 37,38

Methacrylate-based composites face 2 challenges—dental bond strength and marginal microleakage; this was the basis for developing the sandwich technique. However, even with its reduced polymerization shrinkage, LS has not shown a significant improvement in these areas. 28-36 The use of a sandwich technique, therefore, may still be indicated when using LS and the gingival margin is in dentin. The RMGIs utilized in this technique, however, are formulated with a methacrylate-based monomer which may not bond adequately to LS, negating the use of the sandwich technique.

No studies to date have shown whether or not LS can be bonded to RMGI restorative materials, as advocated in the sandwich technique described previously, though the placement instructions for LS do state that “self-adhesive materials such as glass ionomer cements…or resin-modified glass ionomer cements…may be used as cavity liner or reliner.” The purpose of this study is to evaluate the bond strength between an RMGI restorative material (Fuji II LC [FLC], GC America, Inc.) and a methacrylate-based composite (Filtek Z250 [Z250], 3M ESPE) or LS using either the proprietary adhesive (Filtek LS System Adhesive [LSA], 3M ESPE), or a methacrylate-based adhesive (CLEARFIL SE BOND [CSE], Kuraray America, Inc.). This study will also examine the effect of surface preparation—which may occur intraoperatively when using the sandwich technique—on the bond strength.

The null hypotheses to be tested were that there would be no significant differences in the bond strength to FLC based on the type of composite (Z250 or LS), type of adhesive bonding agent (LSA or CSE), or the surface preparation—no treatment (No), bur-roughened (B), etched-only (E), or bur-roughened and etched (Both).

### Materials and methods

The protocol was approved by the Institutional Review Board at Wilford Hall, Ambulatory Surgical Center, Joint Base San Antonio-Lackland, Texas. The materials used in this experiment are FLC, Z250, LS, LSA, and CSE (Table 1). A total of 16 groups were created (Table 2). Ten specimens were prepared per group resulting in 160 total specimens. All samples were created by 1 provider to ensure uniformity of fabrication.

A mold for the RMGI was created using PVC pipe, a washer, and dental stone (Figure). FLC was activated, mixed using a 557 carbide bur (Henry Schein Dental) (Groups 3, 4, 7, 8, 11, 12, 15, and 16), or the surface preparation—no treatment (No), bur-roughened (B), etched-only (E), or bur-roughened and etched (Both). The completed specimens were placed in a sealed container with moistened paper towels to create 100% humidity and stored at 37°C in a lab incubator (Model 20 GC, Quincy Lab Inc.). After 24 hours, the shear bond strength of all specimens was tested using the Instron 5543 testing machine (Instron Corp.) at a crosshead speed of 1 mm/min using the notched blade at a 90 degree angle.

### Table 1. Study materials.

<table>
<thead>
<tr>
<th>Material</th>
<th>Type</th>
<th>Manufacturer</th>
<th>Resin</th>
<th>Filler</th>
</tr>
</thead>
<tbody>
<tr>
<td>Filtek LS</td>
<td>Silorane-based composite resin</td>
<td>3M ESPE</td>
<td>Silorane</td>
<td>quartz, yttrium fluoride</td>
</tr>
<tr>
<td>Filtek Z250</td>
<td>Hybrid methacrylate-based composite resin</td>
<td>3M ESPE</td>
<td>Bis-GMA, Bis-EMA, UDMA, TEGDMA</td>
<td>zirconia, silica</td>
</tr>
<tr>
<td>LS System</td>
<td>Proprietary self-etching primer</td>
<td>3M ESPE</td>
<td>HEMA, Bis-GMA</td>
<td>silane-treated silica</td>
</tr>
<tr>
<td>System</td>
<td>methacrylate-based bonding agent</td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>CLEARFIL</td>
<td>Self-etching primer, methacrylate-</td>
<td>Kuraray America, Inc.</td>
<td>HEMA, Bis-GMA</td>
<td>silanated colloidal silica</td>
</tr>
<tr>
<td>SE BOND</td>
<td>based bonding agent</td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>Fuji II LC</td>
<td>Methacrylate-based resin-modified</td>
<td>GC America, Inc.</td>
<td>HEMA, TMHEDC, TEGDMA</td>
<td>alumino-silicate glass</td>
</tr>
<tr>
<td></td>
<td>glass-ionomer</td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>Gel Etchant</td>
<td>37.5% phosphoric acid gel</td>
<td>Kerr Corporation</td>
<td></td>
<td></td>
</tr>
</tbody>
</table>

Abbreviations: Bis-EMA, bisphenol-A polyethylene glycol diether dimethacrylate; Bis-GMA, bisphenol-A glycidyldimethacrylate; HEMA, hydroxyethyl methacrylate; TEGDMA, triethylene glycol dimethacrylate; TMHEDC, trimethyl hexamethylene dicarbonate; UDMA, urethane dimethacrylate.
Following testing, the specimens were examined under a 10X microscope to determine the failure mode as either adhesive fracture at the adhesive interface, cohesive fracture in the RMGI, cohesive fracture in the composite, mixed fracture (combination of adhesive and cohesive) in the RMGI, mixed fracture (combination of adhesive and cohesive) in the composite, or mixed fracture through the RMGI, adhesive, and composite.

A mean shear bond strength and standard deviation was determined per group. A 3-way ANOVA was used to evaluate the effect of surface preparation with the 4 levels determined previously (No, B, E, Both), and bonding agent with 2 levels (CSE and LSA), on the shear-bond strength of composite with 2 levels (Z250 and LS) to RMGI ($\alpha = 0.05$).

### Results

A significant difference in bond strength was found based on composite ($P < 0.001$) and bonding agent ($P < 0.001$) but not on surface treatment ($P = 0.699$) with no significant interactions ($P > 0.05$). Z250 (14.50 MPa) had a significantly stronger overall shear bond strength than did LS (7.94 MPa). LSA (13.16 MPa) had a significantly stronger shear bond strength than did CSE (9.28 MPa). The difference is seen more clearly when trying to bond with LS (Chart 1).

A failure within the adhesive layer was seen the majority of the time (68%) with LS regardless of adhesive or surface treatment. For Z250, however, the failures were almost exclusively (90%) cohesive within the RMGI restorative material (Chart 2).

### Discussion

Although the manufacturer’s instructions for LS state that “self-adhesive materials such as glass ionomer cements…or resin-modified glass ionomer cements…may be used as cavity liner or reliner,” the results of this study would advise against such use, regardless of bonding agent.

The first null hypothesis was rejected in this study. It stated that there would be no significant difference in the bond strength to FLC based on type of composite (Z250 or LS). As mentioned earlier, it is arguable whether or not the bond strength to tooth structure of LS is as good as the methacrylate-based composites. No formal published articles could be found that studied this relationship. The following recent studies, though, were reported in *International Association of Dental Research* abstracts. Thalacker et al, Yaman et al, and Giacobbi & Vandewalle showed no significant differences in the bond strength of LS to enamel and dentin compared to various methacrylate-based composites. However, El-Shamy et al and Almeida E Silva et al showed the methacrylate-based resin having significantly stronger bond strengths than did the LS.

There are a few studies that have shown bonding of LS to an RMGI. One study by De Goes et al showed that an RMGI luting cement (RelyX Luting Plus Cement, 3M ESPE), bonded to an already cured sample of LS using LSA, though it was significantly weaker than the bond using LSA and RelyX ARC (3M ESPE) or the self-adhesive cement RelyX Unicem (3M ESPE). However, the fact that the LS was the cured substrate to which the RelyX Luting Plus Cement was applied may yield a different result than trying to bond LS to pre-cured Rely X Luting Plus Cement due to the curing chemistry. A more recent study by Boushell et al showed no

### Table 2. Study groupings.

<table>
<thead>
<tr>
<th>Substrate material</th>
<th>Restorative material</th>
<th>Bonding agent</th>
<th>Surface preparation</th>
<th>Group No.</th>
</tr>
</thead>
<tbody>
<tr>
<td>Fuji II LC</td>
<td>Filtek LS</td>
<td>LS System Adhesive</td>
<td>No modification</td>
<td>1</td>
</tr>
<tr>
<td></td>
<td></td>
<td></td>
<td>Acid etch only</td>
<td>2</td>
</tr>
<tr>
<td></td>
<td></td>
<td></td>
<td>Bur modification only</td>
<td>3</td>
</tr>
<tr>
<td></td>
<td></td>
<td></td>
<td>Bur and acid etch</td>
<td>4</td>
</tr>
<tr>
<td>CLEARFIL SE BOND</td>
<td>No modification</td>
<td>5</td>
<td>Acid etch only</td>
<td>6</td>
</tr>
<tr>
<td></td>
<td>Acid etch only</td>
<td>6</td>
<td>Bur modification only</td>
<td>7</td>
</tr>
<tr>
<td></td>
<td>Bur and acid etch</td>
<td>8</td>
<td></td>
<td></td>
</tr>
<tr>
<td>Filtek Z250</td>
<td>LS System Adhesive</td>
<td>No modification</td>
<td>Acid etch only</td>
<td>9</td>
</tr>
<tr>
<td></td>
<td></td>
<td></td>
<td>Bur modification only</td>
<td>10</td>
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<tr>
<td></td>
<td></td>
<td></td>
<td>Bur and acid etch</td>
<td>11</td>
</tr>
<tr>
<td>CLEARFIL SE BOND</td>
<td>No modification</td>
<td>13</td>
<td>Acid etch only</td>
<td>14</td>
</tr>
<tr>
<td></td>
<td>Acid etch only</td>
<td>14</td>
<td>Bur modification only</td>
<td>15</td>
</tr>
<tr>
<td></td>
<td>Bur and acid etch</td>
<td>16</td>
<td></td>
<td></td>
</tr>
</tbody>
</table>

Figure. A 5/16 inch internal diameter washer was glued inside a 1/2 inch PVC pipe filled with dental stone to create a uniform, flat surface specimen mold.
significant difference in the shear bond strength of LS/LSA to Vitrebond Plus (3M ESPE) versus Z250/Adper Scotchbond SE (3M ESPE). The RMGI in the current study was FLC, an RMGI restorative material rather than a luting cement or liner. Perhaps the difference seen in this study is related to the type of RMGI. More studies could be performed to look at this specifically.

The second null hypothesis was also rejected. It stated that there would be no significant difference in the bond strength to FLC based on type of bonding agent (LSA or CSE). The failure mode showed that most of the failures in the LS group were adhesive in nature, whereas the Z250 groups had a high majority being cohesive in nature in the weaker RMGI material. Not only were the failures adhesive, but the LS samples were associated with lower bond strength and larger standard deviations, thus a larger coefficient of variability suggesting a weaker interface. This can also be seen in the 4 samples from the LS group which immediately failed upon testing with 1 other sample testing at near zero MPa. The cohesive failures of the Z250 samples indicate the opposite—a more stable interface. This failure at the interface, especially seen in the LS groups bonded with CSE, reflects part of the results seen in a study by Brandt et al which showed that no bond formed between LS and other tested methacrylate-based bonding agents, with the exception of LSA. Since the majority of the failures between LS and FLC were adhesive in nature, it is prudent to look at the respective dental adhesives and how they compare.

CSE was chosen specifically for this study based on the fact that it, like LSA, is a bonding agent which utilizes a self-etching primer, also known as 2-step self-etch. CSE has been shown in multiple studies to have consistently stronger bond strengths than other 2-step self-etch bonding agents and is similar to the bond strengths of the 3-step etch-and-rinse agents, with superior clinical longevity. Although LSA is advocated by 3M ESPE to be the only bonding agent to be used with LS, after reviewing the CSE instructions for use and LS product profile, very few differences were noted.

At a macro level, the differences noted are limited to a few steps in their application method. The LSA primer is applied for 15 seconds versus CSE’s 20-second application, and the LSA primer is cured prior to adhesive application whereas the CSE primer is not. Because of the lack of studies comparing these differences, it cannot be known whether or not they have the effect seen in this study of LSA having a significantly better bond strength than did CSE.

At a micro level, the basic chemistry appears fairly similar, with both bonding agents containing methacrylate-based resin monomers and neither containing silorane monomers. They both try to span the gap between the necessary hydrophilic layer closest to the dentin and a hydrophobic resin composite, though LS is claimed to be more hydrophobic than methacrylate-based resins. The differences seem to be the pH and a few of the monomers. LSA has a higher pH (2.7 versus 2.0), and the manufacturer states that their acidic monomer helps “initiate the ring-opening cationic cure of Filtek LS restorative, thus providing chemical bonding to Filtek LS.” If it is simply the acidic nature of the monomer, then CSE (which is more acidic) should be able to also initiate the ring-opening action, unless it is too acidic. The main difference in monomers would be CSE’s 10-methacryloxyloxydecyl dihydrogen phosphate (MDP) versus the LSA’s Vitrebond copolymer, though both are stated to have the same purpose of bonding to dentin. Mine et al summarized it best when they stated, “further details on how this methacrylate-based SSA-Bond (referring to LSA) links to the silorane composite is currently not known...” This study demonstrated that both bonding agents could be used to bond Z250 to the RMGI. In fact, LSA outperformed CSE. This agrees with abstracts by De Goes et al and Brandt et al which showed the ability of LSA to bond to other methacrylate-based restorative materials. These results would refute the statement by 3M ESPE that LSA should only be used with LS.

This study failed to reject the third null hypothesis. It stated that there would be no significant difference in the bond strength to FLC based on surface preparation (No, B, E, Both). The results of surface treatment modifications are consistent with previous studies. Bona et al demonstrated that etching did not lessen microleakage between an RMGI and a composite. Maneenut et al conducted a study in which 2 different RMGI cements were
repaired, and the surfaces were treated either with or without etching. No significant differences were noted in bond strength. Also, the SEM they performed “showed little effect from acid treatment.” Roughening the surface with a bur was thought to potentially create a better bond due to increased surface area, but again the results of this study did not substantiate that theory. It does demonstrate, though, that removing the air inhibited layer or accidentally etching the RMGI surface, as may be done clinically during the sandwich technique, does not worsen the bond.

This study tried to determine whether or not Filtek LS could be used in a sandwich configuration, which is used clinically to improve the dentinal margin integrity and decrease its microleakage, possibly reducing the incidence of recurrent caries. The results of this study would not support using this technique with Filtek LS.

Conclusion
Surface modifications did not affect the shear bond strength of the silorane or methacrylate composites to the RMGI. The new silorane composite (Filtek LS) had significantly lower bond strength to the RMGI compared to the methacrylate composite (Filtek Z250). The new silorane adhesive agent (Filtek LS System Adhesive) had significantly higher bond strength to the RMGI compared to the methacrylate adhesive agent (CLEARFIL SE BOND). The greatest bond strengths to the RMGI were produced when using the silorane adhesive agent with the methacrylate composite.

Based on the results of this study, the authors’ recommendations are: the surface modification of the RMGI is not needed to increase bond strength nor does it worsen it; the Filtek LS System Adhesive should be used with Filtek LS; the sandwich technique should not be utilized with Filtek LS based upon its poor bond to an RMGI (Fuji II LC); and the Filtek LS System Adhesive can bond to methacrylate-based composites.

Further studies could confirm these results by performing microleakage tests at the interface of RMGI and Filtek LS versus the interface of RMGI and Filtek Z250.

Author information
Maj. Nuttall is the Deputy Program Director of the 1-Year Advanced Education in General Dentistry Residency, Eglin AFB, Florida. Col. Vandewalle is the director, Dental Research, and Lt. Col. Casey is a training officer of the 2-Year Advanced Education in General Dentistry Residency, Air Force Postgraduate Dental School, Wilford Hall Ambulatory Surgical Center, Joint Base San Antonio-Lackland, Texas, and Uniformed Services University of the Health Sciences, Bethesda, Maryland. Dr. Sabey is the program director, Advanced Education in Endodontics, Louisiana State University School of Dentistry, New Orleans.

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Manufacturers

GC America, Inc., Alsip, IL
800.332.7063, www.gcamerica.com

Henry Schein Dental, Melville, NY
800.372.4346, www.henryschein.com

Instron Corp., Canton, MA
800.877.6674, www.instron.com

Ivoclar Vivadent, Inc., Amherst, NY
800.533.6825, www.ivoclarvivadent.us

Kerr Corporation, Orange, CA
800.537.7123, www.kerrdental.com

Kuraray America, Inc., New York, NY
800.879.1676, www.kuraraydental.com

Quincy Lab Inc., Chicago, IL
773.622.2428, www.quinyclab.com

Ultradent Products, Inc., South Jordan, UT
888.230.1420, www.ultradent.com

3M ESPE, St. Paul, MN
888.364.3577, solutions.3m.com

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