In Vitro Shear Testing of Orthodontic Bonding to Lithium Disilicate Ceramic

Lindsey K. Lyons, DDS, MS  
Jeryl D. English, DDS, MS  
Joe C. Ontiveros, DDS, MS  
Harry I. Bussa Jr., DDS, MS  
Lacy M. Harris, DMD, MSD  
Stephen Laman, DDS  
F. Kurtis Kasper, PhD

Abstract
This article discusses a study to evaluate bond strength of orthodontic brackets to lithium disilicate with varying storage times and surface conditions. Using a phosphate-based orthodontic primer, brackets were bonded for 24 hours and 6 months to ceramic tiles pretreated with hydrofluoric acid, phosphoric acid, or sandblasting. Bond strengths ranged from 12.01 ± 2.64 and 21.83 ± 10.38 MPa, and 23.58 ± 6.70 and 26.76 ± 8.71 MPa for the 6-month and 24-hour groups, respectively. Extended storage showed greater bond strengths than the 24-hour groups. These results led investigators to conclude that orthodontic bracket bonds exceeded the threshold of 6 to 8 MPa for clinical acceptability for all groups using a phosphate-based primer.

Key Words: orthodontic, bond, strength, materials, enamel
The routine use of HFA or mechanical roughening via air abrasion may no longer be necessary to reach clinically acceptable bond strengths when treating orthodontic patients.

Learning Objectives

After reading this article, the participant should be able to:

1. Better understand bonding to lithium disilicate.
2. Streamline his or her bonding technique when bonding to ceramics.
3. Understand the clinical requirement for bond strength when bonding an orthodontic bracket to porcelain.

Disclosures: The authors did not report any disclosures.
Introduction
In a 2015 survey conducted by the American Association of Orthodontists, the number of adult patients seeking orthodontic treatment increased 14% from 2010 to 2012, and increased from 21% in 2012 to 27% in 2014.1,2 Bonding brackets to enamel is routine in modern orthodontics and has become more efficient and effective over time.3,4 However, the majority of these adult patients are presenting with restored dentitions, posing greater challenges for clinicians in terms of bonding to emerging esthetic glass ceramic materials, such as lithium disilicates.5

The application of specialized primers following sandblasting (SB) or hydrofluoric acid (HFA) surface conditioning is used to improve the bond strengths of orthodontic brackets to restorative materials.6,7 A relatively new one-step phosphate-based primer marketed for bonding orthodontic brackets (Assure Plus, Reliance Orthodontic Products; Itasca, IL) claims to be suitable for enamel and all types of restorative materials without the need for sandblasting or HFA surface conditioning. This would reduce the number of clinical steps needed for bonding and may eliminate patient and operator exposure to HFA, both welcome benefits to the clinical protocol.

The aim of the present study was to determine the potential differences in shear bond strength of orthodontic brackets bonded to enamel and lithium disilicate surfaces when using a 1-step orthodontic primer combined with 3 different surface treatment conditions after storage durations of 24 hours and 6 months.

Materials and Methods

Treatment Group Divisions
A total of 120 specimens were tested: 30 enamel surfaces (extracted human premolars) and 90 lithium disilicate tile surfaces (IPS e.max, Ivoclar Vivadent; Amherst, NY). The enamel surfaces and ceramic tiles were divided into 2 groups based on storage duration. Group 1 specimens were stored in distilled water for 24 hours at 37°C before testing. Group 2 specimens were stored in distilled water for 6 months at 37°C before testing. These 2 groups were further divided into the following surface pretreatment subgroups prior to bracket bonding:

- hydrofluoric acid and a silane-based porcelain conditioner
- phosphoric acid and a silane-based porcelain conditioner
- sandblasting and a silane-based porcelain conditioner.

Ceramic Tile Fabrication
Lithium disilicate ceramic ingots (B1, IPS e.max) were received from the manufacturer and fabricated into tile forms (10 x 7 x 3 mm) using the lost-wax technique. Completed pressed tiles were glazed (IPS e.max Ceram), refired, and polished with rubber polishing discs.

Specimen Preparation
All specimens were mounted in cold-cure acrylic (Buehler; Lake Bluff, IL) prior to surface pretreatments. To ensure that no acrylic leaked under the testing surface, a thin, uniform layer of wax was used to secure the specimens in place while the acrylic was added to the mounting cups. The final product was an acrylic cylinder with each specimen securely embedded inside, except for its exposed testing surface where the bracket was to be bonded (Fig 1).

Surface Pretreatment
Before orthodontic brackets were bonded, the exposed surface of all specimens was prepared according to its pretreatment subgroup conditions. All materials used, as well as their specified instructions, are listed in Table 1. Enamel (control) samples were etched for 15 seconds with 37% phosphoric acid (Gel Etchant, Reliance), rinsed with water for 15 seconds, and air-dried with oil-free compressed air. Specimens in Subgroup 1 were etched with 9.6% hydrofluoric acid (Porcelain Etch, Reliance) for 4 minutes, rinsed with water for 30 seconds, and dried with oil-free compressed air. Specimens in Subgroup 2 were etched for 1 minute with the 37% phosphoric acid, rinsed with water for 30 seconds, and air-dried with oil-free compressed air. Specimens in Subgroup 3 were sandblasted with 50-µm aluminum trioxide (Reliance) in a perpendicular orientation at a distance of 10 mm for 3 seconds with an intraoral sandblaster (Etchmaster, Reliance). After the initial surface pretreatment protocol, each subgroup had a thin layer of silane (Porcelain Conditioner, Reliance) applied on the exposed surface, which was allowed to dry for 1 minute. At this time, an application of a phosphate-based orthodontic primer (Assure Plus) was placed in a thin layer on the surface and lightly air-dried for 2 seconds with oil-free compressed air.
Bracket Bonding
All 120 of the bracket specimens were applied in the same manner. A metal mandibular incisor bracket with 80-gauge mesh (American Orthodontics; Sheboygan, WI) was bonded to the pretreated surface with composite resin paste (Transbond XT, 3M Unitek Orthodontics; St. Paul, MN) using 300 g-force as measured by a mechanical spring scale. Any excess adhesive around the bracket base was removed with a micro brush before light curing from the mesial, distal, incisal, and gingival aspects for 3 seconds each. The light cure was completed with a conventional dental halogen lamp (Ortholux XL 3000, 3M Unitek). A single operator carried out all surface pretreatments and bonding procedures completed in this study.

Storage, Thermocycling, and Shear Bond Testing
Immediately after bonding, Group 1 specimens were stored in distilled water at 37°C for 24 hours, and Group 2 specimens were stored in distilled water at 37°C for 6 months. Samples were removed after storage and thermocycled between 5°C and 55°C for 500 cycles with a dwell time of 10 seconds and transfer time of 5 seconds. Shear bond strength testing was performed with a universal testing machine (Instron; Canton, MA) at a crosshead speed of 0.5 mm/min. The acrylic mounted sample and testing blade were secured to the testing device in such a way that an occlusogingival load could be applied to the bracket, thus producing a shear force at the bracket-sample interface (Fig 2). Shear bond strength values were calculated by dividing the maximum load (Newtons) by the base area of the bracket (mm²).

Statistical Analysis
A generalized linear model was used to evaluate the effects of storage duration and surface pretreatment on the response variable (shear bond strength) using Type II sum of squares. Tukey multiple comparison tests were performed to examine the differences in shear bond strength between the 2 storage durations and among the 4 surface pretreatments. All analyses were performed using R statistical software.

Results
Mean shear bond strength values and associated standard deviations for each pretreatment group are reported in Table 2. Shear bond strength differed significantly for the main effects of surface pretreatment (F3,1 12 = 3.5949, p = 0.016) and storage duration (F1,1 12 = 17.42, p = < 0.001), with no significant interaction between them (Fig 3). Strengths ranged from 12.01 ± 2.64 and 21.83 ± 10.38 MPa, and 23.58 ± 6.70 and 26.76 ± 8.71 MPa for the 6-month and 24-hour groups, respectively. Tukey multiple comparisons showed a significant difference between the storage durations (p < 0.001) and that, on average, the shear bond strengths at 6 months were 6.21 MPa higher than those at 24 hours. Tukey multiple comparisons among surface pretreatments showed significant differences between PA and control (p = 0.005) and SB and control (p = 0.006), with no other significant pairwise comparisons.
Discussion
The ideal bond strength for an adequate attachment of orthodontic brackets to enamel is debated in the literature. Traditionally, studies evaluating bond strength to ceramics used a range of 6 to 8 MPa as a sufficient goal for clinical application. More current literature suggests a slightly broader range of 4 to 10 MPa as the in vitro bond strength considered acceptable for clinical use. Since there are not any specific recommendations for bonding to restorative ceramics at this time, it is rational to assume that strengths at least as high as those recommended for enamel are an adequate target. In the present study, 6 to 8 MPa was used as the value at which a reasonable clinical bond would be achieved.

Advantages of PA over HFA and SB
Results from this study revealed that all samples yielded shear bond strengths exceeding that of the recommended 6 to 8 MPa. All three surface pretreatment groups had mean shear bond strengths that were higher than those of the controls, two of which were significantly higher on average: namely, PA samples and SB samples. When comparing the three surface pretreatment groups to one another, there were no significant differences seen. There are several important clinical implications of this finding, as follows:
• Eliminating the use of HFA in the orthodontic office is highly beneficial to both the patient and the doctor in terms of safety. HFA is a caustic agent that can cause

---

Table 2: Mean Shear Bond Strengths (MPa) and Associated Standard Deviations.

<table>
<thead>
<tr>
<th></th>
<th>24 Hours (Group 1)</th>
<th>6 Months (Group 2)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Control</td>
<td>12.01±2.64</td>
<td>23.58±6.70</td>
</tr>
<tr>
<td>Sandblasting (SB)</td>
<td>21.77±9.47</td>
<td>24.12±8.77</td>
</tr>
<tr>
<td>Phosphoric Acid (PA)</td>
<td>21.83±10.38</td>
<td>26.76±8.71</td>
</tr>
<tr>
<td>Hydrofluoric Acid (HFA)</td>
<td>18.03±6.52</td>
<td>24.01±9.30</td>
</tr>
</tbody>
</table>

---

Figure 2: Specimen mounting and load orientation.

Figure 3: Plot of mean shear bond strengths (MPa) for the study groups.
harmful burns and must be applied with extreme caution to avoid contact with soft tissues.\textsuperscript{11-13}

- HFA has been shown to cause greater physical or topographical changes to the ceramic surface when compared to PA.\textsuperscript{11,14} This leads to increased time and difficulty for the clinician in polishing the surface back to its preorthodontic treatment condition.
- The application of HFA also leads to increased clinician time. The recommended ceramic etching time when bonding orthodontic brackets is 4 minutes with HFA compared to 1 minute for PA.\textsuperscript{15,16} In a low-volume practice these few extra minutes may go unnoticed, but in a very busy office, the savings could have a considerable impact over time.
- Many orthodontic offices use PA on a daily basis in their bonding protocol. Because of this, no extra inventory in terms of etchant would be needed to successfully bond to ceramic restorations like lithium disilicate.
- The present findings are consistent with a similar study that reported shear bond strengths comparable to those seen with HFA when using PA as the surface pretreatment prior to bonding orthodontic brackets to feldspathic porcelain.\textsuperscript{11}

The results from the present study show that PA is as effective a surface pretreatment as SB when bonding to lithium disilicate, which is the current recommendation for bonding to ceramics with Assure Plus. PA’s advantages include the following:

- While many orthodontic practices use air abrasion regularly, others do not have the equipment available to perform the sandblasting technique. Many offices, however, stock PA for everyday use. This provides a significant advantage for PA use in place of SB when bonding to restorative ceramics.
- Another advantage of PA over SB, as was noted for PA over HFA, is the reduced amount of damage seen on the glaze of the ceramic restoration.\textsuperscript{11} This benefit saves the clinician valuable time when polishing the restorative surface upon debonding.

**Storage Duration**

The present study found that 6 months’ storage duration resulted in greater shear bond strengths on average compared to performance at 24 hours. This could be due to enhanced mechanical properties attributed to increased polymerization over time. This would be consistent with findings in a 2015 study that assessed bisphenol A (BPA) released from orthodontic adhesives. The results of the study showed a negative correlation between BPA release and degree of conversion.\textsuperscript{17} For both groups that used Transbond XT composite for bonding, as was used in the present study, the BPA release increased from day 1 to day 21, after which the levels decreased to a number lower than the initial day 1 value. This implies an increasing degree of conversion at day 21 and beyond.\textsuperscript{17} Similar results were seen in another study that evaluated fracture toughness in aged composites. In that study, specimens were stored in distilled water at 37°C for either 2 months or 14 months before testing fracture toughness. An increase in fracture toughness was observed for all 4 of the composites that were aged for 14 months.\textsuperscript{18}

From a clinical perspective, the current findings suggest that with the use of Assure Plus as the selected bonding agent, any of the bonding protocols are acceptable to achieve an adequate bond strength when bonding brackets to lithium disilicate restorations. The routine use of HFA or mechanical roughening via air abrasion may no longer be necessary to reach clinically acceptable bond strengths when treating orthodontic patients.

**Conclusions**

Within the limitations of this in vitro study, the following conclusions were drawn:

- Storage for 6 months significantly increased shear bond strengths over values observed at 24 hours.
- There is no significant difference in shear bond strengths attained with the three surface pretreatment protocols (PA, SB, and HFA) when bonding orthodontic brackets to lithium disilicate with Assure Plus and Transbond XT.
- Surface treatment with PA or SB produced significantly higher shear bond strengths than those seen with the orthodontic brackets to enamel controls, possibly eliminating the need for HF application on lithium disilicate.

**Acknowledgments**

The authors thank Dr. J. Nathaniel Holland III (Houston, TX) for his assistance with statistical analysis in the preparation of this article, and Dr. Sudarat Kiat-amnuay (Houston, TX) for material support.

Shear bond strength differed significantly for the main effects of surface pretreatment and storage duration, with no significant interaction between them.
References


Since there are not any specific recommendations for bonding to restorative ceramics at this time, it is rational to assume that strengths at least as high as those recommended for enamel are an adequate target.