To my wife and all who made this milestone possible
ACKNOWLEDGMENTS

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The objective of this study was to determine whether similar repair approaches should be used for different all-ceramic crowns. To perform such an evaluation, HeraCeram (HC), IPS Empress Esthetic (IE), Procera® Crown Alumina (PC), and Cercon® Zirconia (CE) were compared. Seventy-two specimens of HC, IE, PC, and CE were fabricated, embedded in self-curing methylmethacrylate, then grounded flat with sandpapers (240, 400 and 600 grits) until one surface was completely exposed and smooth. Each material was divided into three groups. Each group received surface treatments of: (F) finished only, no additional treatment received, (AC) acid etching with 9.6% hydrofluoric acid, and (SB) sandblasting with 50 µm Al₂O₃. Each of these groups was divided into 4 subgroups. Each subgroup was bonded to composite cylinders using either Clearfil Ceramic Primer (CE), silane and Optibond FL Adhesive (SO), Calibra and XP Bond (SXP ), or 904 Zirconia adhesive with Optibond FL Adhesive (ZR) and then stored 7 days in water at 37°C before shear strength tested.

HC ceramic performed better on both etched and sandblasted surfaces than finished surfaces. Of the three adhesive systems, CE gave the highest mean strength values independent of surface treatments. There was no significant difference in bond strength between etched and sandblasted surface in any adhesive systems. The IP results revealed that surface etching with
9.6% hydrofluoric acid gave the highest shear bond strength of the other treated groups. There was no significant difference in CE performance when compared to etched or sandblasted SO, or etched SXP adhesive systems. PC had the highest shear bond strength when the surface had been sandblasted. CE and SXP showed no significant differences when it come to surface treatments. For CZ, sandblasting gave the highest shear bond strength, and when it comes to adhesive systems on CZ, SXP appeared to have the highest bond strength independent of surface treatment. It can be concluded from this study that in order to repair a fractured or chipped all ceramic crown, one needs to know the composition of the core ceramic structures.
CHAPTER 1
INTRODUCTION AND LITERATURE REVIEW

Introduction

During the past 10 to 20 years, patient demand for esthetic and metal-free restorations and the use of “all-ceramic” crown/bridge restorations have increased. As a result of the increased use of these ceramic materials, the number of failures can also be expected to increase because of dentists’ and technicians’ attempts to achieve esthetic design, particularly of complex multi-tooth bridges, where the ceramic materials cannot withstand the required loading conditions. Other failures result because of high localized stresses generated by hard particulates caught between the teeth during chewing. Poor adhesion between the ceramic restoration and the underlying tooth is an additional cause for failure.

Today, the greatest shortcoming with all-ceramic dental restorations is the tendency to chip or fracture. To decrease the fracture risk, ceramics such as alumina and zirconia are now used, which provides superior fracture toughness compared to more traditional dental ceramics (Figure 1-1). However, despite the superior fracture toughness of alumina and zirconia, it is unlikely that the use of these materials will decrease the risk for chipping of the outer layer of the restoration as long as the entire restoration is not made from these ceramics. This is because both alumina and zirconia are too opaque and whitish in color to serve as ideal aesthetic materials. Therefore, to overcome the problem with their aesthetic limitations, these materials are used as core substrates and as such, are usually covered with layers of feldspathic veneering porcelain with superior aesthetic properties.

Unfortunately, these outer ceramic layers have properties similar to those used in dentistry for the past fifty years. Consequently, it seems reasonable to suspect that even though fractures of the ceramic core-structures may decrease as a result of using alumina and zirconia, the
tendency for chipping in the veneering layer should remain the same. Based on available information, it is known that failures resulting from ceramic fractures range from ~ 2 to 4% for ceramic fused-to-metal crowns between seven and ten years old, while with an Allceram crowns, the failure rate between five and ten years is ~ 2% and 6.5% respectively.

Replacement of chipped ceramic restoration is not always necessary. The most practical solution, considering the replacement cost of an additional tooth structure and additional trauma to the tooth during the removal of a core structure, is to repair the crown. As a result, in determining the ability to chair-side repair chipped or partly fractured ceramic restorations, it is important to consider reparability when an all-ceramic dental material is being selected.

**Repairing a Chipped Ceramic Restoration**

The treatment of a chipped ceramic restoration depends on the size of the chip. In some cases with smaller degrees of chipping, the only treatment possibly needed is to smooth and finish the sharp edges of the fractured surface. If the fractured surface is isolated to the outer ceramic or if the core material has been exposed, it can be repaired with a composite resin bonded to the ceramic surface. This type of repair consists of three steps: A) surface roughening, B) placement of coupling agent, and C) placement of composite resin.

**Surface Roughening**

To succeed with such a repair, the ceramic surface is etched with a hydrofluoric acid and rinsed. If the exposed ceramic surface is not acid resistant, the acid removes the least acid-resistant phases of the ceramic and creates an irregular and rough surface. The rough surface formed by acid etching facilitates the formation of micromechanical retention of composite resin to the ceramic surface.

In addition to hydrofluoric acid etching, air abrasion with 50-µm aluminum oxide (Al₂O₃) particles under air pressure is another method of surface roughening. The impact from the
abrasive particles knocks away the weaker phases of the ceramic and creates an irregular and rough surface, which increases the bondable surface area. The roughness, however, in contrast to the etched surface, is not associated with true micromechanical retention, as sandblasting causes an overall surface chipping while etching results in micro-cavity formation.22

**Coupling Agents**

The etched or sandblasted surface is then silane coated and dried.22,23 The silane treatment forms covalent bonds to the ceramic surface 24-26 and to the methacrylate groups of the methacrylate-based composite resin molecules (Figure 1-2). Also, it enhances the composite resin wetting of the ceramic surface.27-29 A regular bonding resin is placed on the silane-coated surface and cured, whereupon the composite is placed and cured. If, however, a core structure such as alumina or zirconia is exposed, the bonding process becomes more complicated because hydrofluoric acid is not capable of forming a rough alumina or zirconia surface.30 Besides, the silane may not act as an efficient coupling agent on those surfaces as it is on regular silica-based ceramic surfaces.

**Composite Resin Application**

When a chipped ceramic surface repair uses a composite resin, thermal expansion coefficient and modulus of elasticity are important properties to consider. The benefit with low thermal expansion coefficient is that it better matches the ceramic, while a lower modulus results in lower stress levels introduced during curing. In addition, a lower modulus also decreases the stress level induced in that particular region if one assumes that the region deforms a certain amount during chewing, independent of composite (e.g., primary contact in occlusion). Unfortunately, thermal expansion coefficient and modulus of elasticity are opposite properties to each other. Low thermal expansion is associated with high modulus and vice versa. Besides, low modulus also is associated with lower filler fraction and higher polymerization shrinkage.
Because of the latter, a safer option is to move toward a composite with higher filler fraction and thus higher modulus and lower thermal expansion coefficient. Unfortunately, no clinical data is available today determining the superiority of any particular composite type, and as a consequence, no simple selection process exists. Perhaps the most important considerations are the aesthetic appearance and the ease of manipulation.

Some in vitro studies have tried to address the selection of composite for ceramic repairs. Accordingly, the bond between the ceramic surface and the composite restoration must be sufficiently strong to withstand the functional loads. Therefore, a minimal coefficient of thermal expansion and polymerization shrinkage are considerations when choosing the repair materials. Bond strength is also dependent on the type of the composite resin used. According to Gregory and Moss (1990), repairs with larger particle-sized composite, hybrid type at the porcelain interface and overlaid with micro-filled composites resulted in higher bond strength values than when a homogeneous small particle-sized composite was used. Hybrid composite resin increases strength and decreases stress compared with a micro-filled composite. If a micro-filled composite resin is used, the loaded restoration should not be exposed to fatigue loading.

**Commonly Used Ceramic Materials in Dentistry**

The success of repairing chipped ceramics depends on the surface treatment of the ceramic surface as well as on the coupling agent (silane or adhesive) used. In addition, the composition of the ceramic affects the surface treatment as well as the way a coupling agent interacts with the ceramic surface. The following section focuses on four commonly used ceramic systems. These systems include the traditional feldspathic ceramics, pressable leucite-based ceramics, alumina-based ceramics, and zirconia-based ceramics.
Feldspathic Ceramic

The Chinese were the leaders in the development of the ceramics materials in the seventeenth and eighteenth centuries. The composition of these ceramics was around fifty percent kaolinite, 25 percent feldspar, and 25 percent quartz. The first single-tooth ceramic material was introduced to dentistry in the 1880s and was used to make full porcelain jacket crowns and porcelain inlays. The compositions of these early dental ceramics were very close to the composition of the Chinese porcelains which were rich in mullite (Al₆Si₂O₁₃). Another name for mullite is porcelainite, from which the word porcelain originated.

Over the years, the compositions of dental ceramics have changed. Mullite and free quartz were removed, while the Na₂O, K₂O and the leucite (AlSi₂O₆) contents were increased. The change in the composition improved the translucence and the strength of the material.

Leucite-Reinforced Ceramic (Hot-Pressed Systems)

Leucite-reinforced ceramic was developed by Wohlwend in 1983 at the Dental Institute, Zurich University. In 1986, Ivoclar Vivadent bought the patent and presented it to the market as IPS Empress in 1990. In this system, leucite crystals measuring 1-5 μm is added to the dental ceramic to enhance flexural strength and fracture resistance. The leucite crystals reinforce the material by preventing crack propagation and failure. Also, by heat-pressing the ceramic, large pores are avoided. Heat-pressing also promotes a good dispersion of the crystalline phase within the glassy matrix, and it reduces the amount of ceramic shrinkage, which results in higher flexural strength.

High-Alumina Reinforced Ceramics

High-purity alumina copings were described by Andersson and Oden in 1993. This system was marketed as the Procera All-Ceramic System (Procera-Sandvik, Stockholm, Sweden). Procera all-ceramic copings are manufactured by using a dry-pressing technique
against enlarged models of the tooth preparation to compact a high-purity alumina powder (Al₂O₃ > 99.9%). The enlarged dies are made by the Procera® system, which utilizes a computer-aided manufacturing (CAM) and computer-aided design (CAD) technology to mill the dies used to press the Al₂O₃ framework. After the frameworks are pressed, they are heat processed and shrunk to their final size. The Al₂O₃ structure has no glassy phase between their particles; however, because of the far-from-ideal aesthetic properties of Al₂O₃, the core structure is veneered with an aesthetic ceramic. This process is completed by firing feldspathic veneering porcelains such as NobelRondo™ Alumina onto the alumina core to provide the color and form of the restoration.

**Zirconia Reinforced Ceramics**

During the past few years, several different zirconia-based core products were introduced. One such system is the Cercon Zirconia System, which like Procera, uses a CAM/CAD process. After the tooth has been prepared and an impression and a gypsum model made, a wax reconstruction is made on the die. The wax model placed on the gypsum model is transferred to the Cercon Ceramic System, in which the model is scanned with and without the wax pattern. The computer then analyzes the two scans and constructs a three-dimensional model of the wax pattern. The coordinates of the model are transferred to a milling unit, and a core structure is milled in partly-sintered zirconia. In order to produce a dense structure, the partly-sintered zirconia structure is transferred to a computerized oven in which the core finally is sintered. During the sintering process, the core structure shrinks. To overcome the shrinkage, the milled framework must be oversized to a size determined by the computer when the scanned information is processed.

In addition to Cercon, zirconia frameworks are made by systems such as Lava and Denzir. Lava uses similar technology to Cercon, while Denzir mills the core structure directly from
industrially processed and sintered zirconia. Thus, in the case of Denzir, no sintering is performed after the milling process is completed. However, independent of which process is used, the aesthetic properties of zirconia are not ideal, and therefore, a veneering ceramic is used to finalize the ceramic reconstruction, like in the case of Al₂O₃ cores.

**Novelty, Scope, and Goal of Study**

Because of an increased use of different all-ceramic restorations, it is predicted that the interest in repairing such restorations will increase in the future. It is expected that clinicians will find themselves in situations where they are unsure of which ceramic system they are repairing. Such situations will be common in states such as Florida, where patients received their restorations in other states at a younger age and then retired in Florida. Under such conditions, if a ceramic failure occurs, the dentist in Florida may not have access to information about the ceramic system used when the ceramic restoration is made. In such a situation, how should the dentist treat the ceramic surface and pick a coupling agent with the highest likelihood of being successful? Also, if the most likely treatment is picked, which ceramic system can cause the biggest problem? Furthermore, if the dentist happens to know the ceramic type, which treatment should then be used to optimize the outcome?

To address these questions, a comparison of composite repair strength of different ceramics after they have been surface treated and then bonded with a composite was arranged. The ceramic materials tested in this study were regular feldspathic ceramics, pressed ceramics, alumina, and zirconia. The ceramic materials were surface treated with either 9.6 percent hydrofluoric acid, sandblasted, or finished before composite cylinders were bonded with four different adhesive systems to these surfaces.

The hypothesis is that the best bonding would occur to ceramics that responded most to the hydrofluoric acid treatment. By acid etching the surface, it should be possible to selectively
dissolve less acid resistant phases and thereby form micro-mechanical retention sites. The hydrofluoric acid treatment was hypothesized to work best on feldspathic and pressed ceramics because studies by Borges et al. 2003 have shown that alumina and zirconia do not respond well to such an acid treatment. In addition to hydrofluoric acid etching, surface roughening can also be achieved by sandblasting the ceramic surface. During sandblasting, microchips are knocked away from the ceramic surface as a result of the impact from the abrasive particles. Air abrasion was hypothesized to be the optimal surface treatment to roughen zirconia and alumina compared to hydrofluoric acid. In order to determine whether etching or sandblasting would perform best, a comparison of these two treatments with finished surfaces serving as controls was used.

When it came to comparing feldspathic and pressed ceramics with alumina and zirconia, the hypothesis was that sandblasted feldspathic and pressed ceramics would probably provide better bonding conditions. The reason was that due to the lower fracture toughness of feldspathic and pressed ceramics, these two ceramics would respond more extensively to sandblasting than alumina and zirconia. Of the investigated ceramics, the zirconia-based core material has the highest fracture toughness—twice as high as the second toughness ceramic investigated, the alumina core material. Thus, the higher fracture toughness of alumina and zirconia (Figure 1-1) suggests that these two ceramics would not microchip as much as the feldspathic and pressed ceramics during the sandblasting process.

Regarding the use of different adhesives, the hypothesis was that the similarities between the silica structure and the silane molecules would result in better compatibility bonding between the silane and the feldspathic and pressed ceramics with their SiO2 content in contrast to the alumina and zirconia ceramics with their Al2O3 or ZrO2 groups. Even though silane treatment enhances bonding, such a treatment may not contribute as much to retention as micromechanical
retention. The reason may simply be that only a maximum of one-third of all SiOH groups present on a silica surface interacts with the silane molecules.\textsuperscript{26} Such a limited chemical bond formation supports the assumption that the hydrofluoric-etched or sandblasted feldspathic and pressed specimens would form stronger composite bond strength values than the smoothest surfaces. If silane is the key bond mechanism, silane-treated finished specimens would perform as well as etched or sandblasted. In fact, both etching and sandblasting would increase the probability for the introduction of defects at the interface, suggesting that a reliable silane bonding on a finished surface should perform better than a silane bonding on a defect rich surface. Thus, it is hypothesized that because of the 1/3 SiOH interaction, silane treatment is just a supplement to an etched or sandblasted surface, and by comparing the overall results, it is expected to find the best adhesion to surfaces with the highest micromechanical retention ability. In this case, this would be etched surfaces followed by sandblasted surfaces with the lowest bond strength values being the finished surfaces.

To summarize, it was hypothesized that:

a) Hydrofluoric acid treatment would work best on feldspathic and pressed ceramics.

b) Air abrasion would provide better surface roughening on alumina and zirconia ceramics, and therefore provide better bonding on these two ceramics than hydrofluoric acid.

c) Regarding the use of different adhesives, including the silane treatment, the similarities between the silica structure and the silane molecules would result in better bonding between the silane and the feldspathic and pressed ceramics than with the Al\textsubscript{2}O\textsubscript{3} and ZrO\textsubscript{2} ceramics.

d) Because only 1/3rd of the SiOH groups on a silica surface react with silane, silane treatment is just a supplement to an etched or sandblasted surface. Consequently, it was expected that the best adhesion would occur to surfaces with the highest micromechanical retention ability. Thus, it was expected that etched surfaces should be followed by sandblasted surfaces, while the lowest bond strength values would be found for the finished surfaces.
Figure 1-1. Fracture toughness of ceramic materials.\textsuperscript{46}

Figure 1-2. The formation of covalent bond between the silica surface and the silane (Courtesy by K-J Söderholm).
CHAPTER 2
MATERIALS AND METHODS

Ceramic Materials and Their Preparation

Four ceramic materials—feldspathic (HeraCeram [HC], lot # 1602, Heraeus Kulzer, Inc. Armonk, NY, USA), leucite-reinforced glass (IPS Empress Esthetic Rohling [IE], batch JM0639, Ivoclar Vivadent, Schaan, Lichtenstein), aluminum oxide (Procera® Crown Alumina [PC], Nobel Biocare, Sweden), and yttria-stabilized zirconia (Cercon® Zirconia [CZ], lot # 20020089, DeTray, Konstantin, Germany)—were prepared as follows. The HC samples consisted of 72 ceramic blocks, ~ 5 x 5 x 5, and were made by Precision Dental Lab, Gainesville, Florida, USA. The manufacturer of IE donated sixteen pressed cylinders, ~ 25 mm long with a diameter of 5 mm. From these cylinders, 72 cylinders, ~ 5 mm long, were cut. The 72 PC samples were donated several years ago by the manufacturer of PC. These samples consisted of disks, 16.4 mm in diameter and 1.5 mm thick. The manufacturer of CZ provided 72 processed cylinders of CZ used to evaluate their product. These cylinders were ~ 8 mm in diameter and ~ 5 mm long. All 288 ceramic samples were imbedded in self-curing methylmethacrylate (Technovit 4004, lot # 64708471, Heraeus Kulzer GmbH, Germany) cylinders (30 mm diameter x 30 mm height), with one of the ceramic surfaces exposed. After the methylmethacrylate set, the exposed ceramic surface was ground flat with sandpaper (240, 400 and 600 grits).

The 72 specimens per ceramic group were divided into three groups of 24 specimens each. One of these main groups, the finished group (F), was not processed further before bonding was performed, while the other two groups were acid etched (AE) with 9.6% hydrofluoric acid (Porcelain Etch Gel, Pulpdent Corporation, Watertown, MA, USA) for two minutes and then rinsed with water for thirty seconds just before different bonding procedures were performed or were sandblasted (S) with 50 μm Al₂O₃ for five seconds under a pressure of two bars with the
nozzle held 10 mm away from the ceramic surface just before the different bonding procedures were performed. Each of these main groups was in turn divided into four subgroups, each consisting of six specimens. Each of these subgroups was treated with one of the following bonding procedures.

**Bonding Procedure 1 (CE)**

The ceramic surface was coated with Clearfil Ceramic Primer (lot # 00003A, Kuraray America, Inc. NY, USA) and then dried by blowing mild oil-free air for five second. A stainless still disk, 2 mm thick, 12 mm wide, and with a central hole 3 mm in diameter, was used as a mold for the composite cylinder bonded to the adhesive. The surface of the disk contacting the ceramic surface was covered with a double adhesive tape. The adhesive-coated surface was placed in contact with the flat ceramic-methylmethacrylate surface with the central hole over the ceramic surface only. A small increment of a composite material (Venus, A2, lot# 010118, Heraeus Kulzer GmbH, Germany) was inserted to half the height of the cylinder. The composite then was adapted to the ceramic surface to secure a good composite/adhesive contact and was light-cured (Translux Power Blue, Heraeus Kulzer GmbH, Germany) for twenty seconds. The power density of the light source was ~ 1000 mW/cm² determined with a light meter (Cure Rite, Dentsply Caulk, Milford, DE, USA). After the first increment was cured, a second increment was added to fill the entire mold, and this layer was cured for another twenty seconds.

**Bonding Procedure 2 (SO)**

A ceramic surface was coated with Dry-Rite (Drying Agent, Pulpdent Corporation), and air dried for five seconds, whereupon a silane coupling agent (Calibra, lot # 070215, Dentsply, Caulk) was placed and dried five seconds. One coat of OptiBond® FL Adhesive (lot # 4XX233, Kerr Corporation, Orange, CA, USA) was placed over the silane and lightly air thinned for five
seconds after which it was cured for ten seconds. A composite cylinder was then bonded as
described above.

**Bonding Procedure 3 (SXP)**

The ceramic surface was dried and silane treated as described above, whereupon a coating
of XP Bond (lot # 0609001329, Dentsply, DeTray, Konstantin, Germany) was placed and
allowed to interact for twenty seconds before it was air thinned five seconds and light cured for
ten seconds. Following these steps, a composite cylinder was bonded as described above.

**Bonding Procedure 4 (ZR)**

The adhesive used in this experiment was a zirconia adhesive called 904 Zirconia (lot #
722842, Cotronics Corporation, Brooklyn, NY, USA) and is used within the electronic/electrical
industry. One drop of the 904 adhesive and one drop of the 904 thinner/hardener (lot # 722844,
Cotronics Corporation, Brooklyn, NY, USA) were mixed for fifteen seconds and then placed on
the exposed ceramic surface. The coating was lightly air-thinned for ten seconds and heated to
50°C with a hairdryer for thirty seconds, whereupon one coat of Optibond FL Adhesive was
placed and lightly air-thinned for five seconds after which it was light-cured for ten seconds.
Following these steps, the metal molds were placed as described and filled with composite as
described earlier.

After bonding was completed, the specimens were immersed into tap water and stored for
one week at 37°C before they were tested in shear.

**Shear Bond Strength Testing**

The specimens with the metal rings surrounding the composite cylinders were secured in a
guillotine-like specimen holder attached to a universal testing machine (Model 1125, Instron,
Canton, MA). The blade of the guillotine-like specimen holder was placed on top of the edge of
the metal disk surrounding the bonded composite cylinder and then loaded under the load cell.
with a cross-head speed of 0.5 mm/min (Figure 2-1). At a certain load, the metal disk with its composite cylinder was separated in shear from the ceramic surface. The shear load at failure was then divided by the cross-sectional area of the cylindrical composite sample, providing the shear stress level at failure. The fractured surfaces then were evaluated in an attempt to determine where the failures occurred.

**Statistical Evaluation**

The experimental treatments formed a balanced 3 x 4 factorial design with six replicates per treatment combination. Because the distribution of shear bond strength exhibited strong skewness and a non-constant variance, a natural-logarithm scale was used for the analysis. A paired t-Test with modified degrees of freedom was used; this test is known as the Satterthwaite’s approximation. A P-value less than 0.05 was considered as significant. Because it was clear at this point that the 904 adhesive with Optibond FL Adhesive (ZR) was not working as it failed even during storage in water or placement in the testing device, the ZR group was not included in the final analysis.
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<td>HC</td>
<td>Feldspathic</td>
<td>SiO₂ (60-72%), Al₂O₃ (8-12%), K₂O (11-17%), Na₂O (4-9%), Li₂O (0.3-1.2%), CeO₂ (0.5-3.0%), F (0.1-1.5%), CaO (0.1-3.0), B₂O₃ (0.2-1.2%), SnO₂, ZrO₂, Y₂O₃, Li₂O, Inorganic pigments (0.0-3.5%)</td>
<td>Heraeus Kulzer Inc. Armonk, NY, USA</td>
</tr>
<tr>
<td>IPS Empress</td>
<td>IE</td>
<td>Leucite-reinforced</td>
<td>SiO₂ (63%), Al₂O₃ (17.7%), K₂O (11.2%), Na₂O (4.6%), CeO₂ (1.6%), B₂O₃, CaO, BaO, TiO₂ (1%)</td>
<td>Ivoclar Vivadent, Schaan, Lichtenstein</td>
</tr>
<tr>
<td>Procera® Crown Alumina</td>
<td>PC</td>
<td>High-alumina reinforced</td>
<td>Al₂O₃ (99.5%)</td>
<td>Nobel Biocare, Sweden</td>
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<tr>
<td>Cercon® Zirconia</td>
<td>CZ</td>
<td>Zirconia reinforced</td>
<td>ZrO₂, Y₂O₃ (5%), Hf₂O₃ &lt; 2%, DeTray, Konstantin, Germany</td>
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<p>| Table 2-2. Description of adhesive materials used in study |</p>
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<tr>
<td>Clearfil Ceramic</td>
<td>CE</td>
<td>Ethanol 80-100%, trimethoxysilylpropyl methacrylate &lt; 5%, 10-Methacryloyloxydecyl dihydrogen phosphate (MDP)</td>
<td>Kuraray America, Inc. NY, USA</td>
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<tr>
<td>Optibond FL Adhesive</td>
<td>SO</td>
<td>Uncured Methacrylate Ester Monomers 50-60%, Triethylene Glycol Dimethacrylate 5-10%, Ytterbium rifluoride 12-17%</td>
<td>Kerr Corporation, Orange, CA, USA</td>
</tr>
<tr>
<td>XP Bond</td>
<td>SXP</td>
<td>TCB resin, PENTA, UDMA, TEGDMA, HEMA, Butylated benzenediol (stabilizer), Ethyl-4-dimethylaminobenzoate, Camphorquinone, Functionalised amorphous silica, t-butanol</td>
<td>DeTray, Konstantin, Germany</td>
</tr>
<tr>
<td>904 Zirconia</td>
<td>ZR</td>
<td>Composition was not available from the company</td>
<td>Cotronics Corporation, Brooklyn, NY, USA</td>
</tr>
</tbody>
</table>
Figure 2-1. Shear bond testing device with bonded specimen. Metal ring used to shape the composite was kept in place during the test.
Results

Results of the mean shear bond strength values and standard deviations for the study ceramics groups are shown in Figures 3-1 to 3-4. The significance levels between pairs are shown in Tables 3-1 to 3-11. Figures 3-1 to 3-4 show numerical results for the ZR adhesive, while ZR is not included in the tables because of its inferior results. The ZR results are not included in the tables because of the numerous spontaneous failures during storage in water, suggesting that ZR was so inferior to the other adhesives that there was no need for further analyses of that product.

Feldspathic Ceramic (HC)

Surface treatment: Figure 3-1 and Table 3-1 show that both etched and sandblasted surfaces performed better than finished surfaces. However, regarding separating etching and sandblasting, a certain interaction exists between the surface treatments and the used adhesives, making it impossible to identify one of these two treatments as the one to recommend independent of adhesive selection.

Adhesive materials: Of the three adhesive systems, CE gave the highest mean strength values independent of surface treatments. The highest strength values with CE were for sandblasted surfaces even though it was not significantly higher than the etched surface. Sandblasting gave significantly higher bond strength than with finished surfaces, while no significant difference between etched and finished surfaces was determined (Table 3-1).

The trends were somewhat different for the SO and SXP adhesives. The etched surfaces had the highest bond strength values and the finished surfaces the lowest values for these
adhesives. Table 3-1 shows that there is no significant difference in bond strength between etched and sandblasted surfaces in any adhesive system.

**Summary- HC:** The best treatment for the HC is either sandblasted or etched the surface, and any of the three adhesives can be used (Table 3-1). Because of these findings, the first hypothesis that hydrofluoric acid treatment would work best on feldspathic ceramics could not be supported since sandblasting performed equally well in this type of ceramic. However, the fourth hypothesis that silane treatment is just a supplement to micromechanical retention is supported, because the best adhesion occurred on etched and sandblasted surfaces. The lowest bond strength values were found on the finished surfaces.

**Leucite-Reinforced Ceramic Systems (IP)**

**Surface treatment:** Figure 3-2 and Table 3-2 reveal that surface etching with 9.6% hydrofluoric acid resulted in the highest shear bond strength, followed by sandblasting and then a finished surface. However, it is important to note that interactions exist between surface treatments and adhesives, making it difficult to compare sandblasted and finished surfaces without considering the adhesive used.

**Adhesive materials:** Of the three adhesives, CE resulted in the highest mean shear bond strength value independent of surface treatment. Of the three surface treatments, CE bonded best to etched surfaces and weakest to finished surfaces. However, the differences between these three surface treatment groups were too small to be statistically significant (Table 3-2).

Comparing SXP and SO revealed that both had the highest values when bonded to etched surfaces, while SXP had the lowest value when bonded to sandblasted surfaces, and SO had its lowest value when bonded to finished surfaces. The strength value for the etched surfaces treated with SXP was significantly higher than the sandblasted or finished surfaces. The difference between the sandblasted and finished surfaces treated with SXP was not significant (Table 3-2).
The SO groups had the same pattern as the CE groups, showing the highest strength values for the etched surfaces and the lowest for the finished surfaces. When bonded to etched or sandblasted surfaces, however, SO produced significantly higher values than when bonded to finished surfaces while showing no significant difference between the etched and the sandblasted surfaces (Table 3-2).

**Summary- IP:** The best treatment according to these results is to etch an IP surface with 9.6% hydrofluoric acid. Of the adhesives, CE gave similar results independent of surface treatment, while SXP produced comparably good results to CE when the surface was etched (Table 3-2). However, SXP gave significantly lower values than CE when the surfaces were sandblasted or finished. For this type of ceramic, the first hypothesis that hydrofluoric acid treatment would perform best is weakly supported. The interaction between adhesive and surface treatment is too strong to strongly support that hypothesis. The fourth hypothesis that silane treatment is just a supplement to micromechanical retention is weakly supported. The weak support relates to the performance of CE that contains silane and gave only a non-significant difference between the different surface treatments.

**High-Alumina Reinforced Ceramics (PC)**

**Surface treatment:** Sandblasting resulted in the highest shear bond strength values ($p < 0.05$) (Figure 3-3 and Table 3-3). The other two treatments, etching or finishing, did not differ from each other ($p > 0.05$).

**Adhesive material:** Figure 3-3 shows that SXP always produced the highest shear bond strength values of the three adhesives, independent of surface treatment. Figure 3-3 also determines that SXP had the highest value for the sandblasted surfaces and the lowest values for the etched surfaces. However, no significant difference in bond strength was determined when SXP is used on etched, finished, or sandblasted surfaces, as is depicted on Table 3-3. Figure 3-3
shows that the bond strength values of the sandblasted treatment combined with SXP adhesive were significantly higher than the other groups.

Product CE followed the same pattern as SXP regarding the highest mean strength value for sandblasted surfaces and the lowest for etched surfaces. Also, the differences in strength between the three surface groups were not significant (p > 0.05) (Table 3-3).

Of the three adhesives, the SO adhesive deviated from the other two adhesives considering surface treatments and bond strength values. The trend for SO was to have the highest shear bond strength for the sandblasted surface and the lowest for the finished surface, a difference that was significant. The SO adhesive gave the lowest shear bond strength values for PC treated with either etching or finishing treatment.

**Summary –PC:** The best treatment according to these results is to sandblast PC and use SXP or CE as the adhesive system (Table 3-3). The second hypothesis, suggesting that air abrasion would provide better surface roughening on alumina and therefore would provide better bonding than hydrofluoric acid was supported. The third hypothesis suggesting that due Al₂O₃ (or ZrO₂) surfaces would be less compatible with the silane than a SiO₂ surface is supported. All the adhesive contained silane, but in contrast to SO, both SXP and CE contained other active coupling agents. It seems as these active component improved the adhesion.

**Zirconia Reinforced Ceramics (CZ)**

**Surface treatments:** Figure 3-4 and Table 3-4 show that sandblasting produced the highest shear bond strength (p < 0.05). The other two treatments, etching or finishing, did not differ significantly from each other (p > 0.05).

**Adhesive material:** Figure 3-4 shows that SXP, independent of a surface treatment, always resulted in the highest shear bond strength value of the three adhesives. Figure 3-4 also reveals that SXP has the highest value for the sandblasted surfaces and the lowest value for the
etched CZ surface. However, by looking at Table 3-4, there was no significant difference in shear bond strength between the etched or finished surfaces when SXP was used, while SXP in combination with sandblasted surfaces resulted in significantly higher shear bond strength values than the other two surface treatments ($p < 0.05$). The result of Figure 3-4 shows that the bond strength of a sandblasted treatment combined with SXP adhesive was significantly higher than the other groups.

Adhesive CE followed the same trend as SXP regarding the highest mean strength value for sandblasted surfaces and the lowest for etched surfaces (Figure 3-4). However, that trend was not significant because there were no significant differences in strength between the three surface groups ($p > 0.05$) (Table 3-4).

Of the three adhesives, SO deviated from the other two adhesives considering surface treatments and bond strength values. The trend for SO was to have the highest shear bond strength for the sandblasted surface and the lowest for the finished surface, a significant difference (Table 3-4). The etched surface, though, was not significantly stronger than the finished surface and not significantly weaker than the sandblasted surface.

**Summary – CZ:** The best treatment according to these results is to sandblast CZ and use SXP or CE as the adhesive system (Table 3-4). Also for CZ, the second hypothesis, suggesting that air abrasion would provide better surface roughening on alumina and therefore would provide better bonding than hydrofluoric acid was supported. The same was true for the third hypothesis, suggesting that Al$_2$O$_3$ and ZrO$_2$ surfaces would be less compatible with the silane than a SiO$_2$ surface, was supported. The two adhesives that contained other active components than silane (SXP and CE) performed better than the adhesive system that just relied upon silane (SO).
Modes of Failure

Almost all failures were adhesive in nature. Only IP and HC failed cohesively, and these failures occurred mainly in the surface-etched groups even though some failures could also be seen in the sandblasted groups (Figures 3-5 to 3-8). Not a single failure in the ceramic was noticed for any of the CZ or PC specimens, independent of surface treatment (Figure 3-9).

Discussion

Statistical Evaluation

Most studies similar to this study use one-way, two-way, or three-way ANOVA. However, these results showed to have standard deviations between the different groups too large to make such an approach suitable. In order to run a reliable ANOVA evaluation on this data, more than six specimens per experimental group were needed as well as standard deviations of similar sizes. Because the key factor regarding bonding is to avoid failures, we felt that low strength values were of greater importance than the highest values. Because of that as well as the skewed distribution, the statistical evaluations were performed on the logarithm of the strength values. By using such an approach, the low values could be emphasized on the expense of the highest strength values. However, in Figures 3-1 to 3-4, decimal numbers were used to present the experimental results. The reason is simply that such a presentation helps the reader to understand the magnitude of the values generated for the different groups.

Surface Treatment

To better understand the effects of the different surface treatments, it is important to look at the different ceramics and their compositions. As seen from Table 2-1, there is a clear difference between the four ceramics. The HC and IP ceramics contain many different components, while PC and CZ ceramics are primarily comprised of either Al₂O₃ or ZrO₂ with some yttrium. It is often assumed that hydrofluoric acid does not etch Al₂O₃ or ZrO₂ in contrast to ceramics such
as feldspathic porcelain and leucite. Review of the literature reveals that Al₂O₃ and ZrO₂-based ceramics are acid resistant and perform better when their surfaces are sandblasted. 30, 35, 48

By etching feldspathic ceramic with 9.6% hydrofluoric acid, the acid dissolves the glassy phase of the feldspathic ceramic, which results in a micro-undercut formation at the surface of the ceramic. These micro-undercuts are then filled by the adhesive resin, resulting in micromechanical interlocking within the ceramic. 22, 49

Leucite in the IPS Empress ceramic showed a morphological surface change when treated with 10% hydrofluoric acid. 30 Etching the HC and IP groups with 9.6% hydrofluoric acid resulted in the highest bond strength values. This finding is in agreement with the earlier works. 22, 50, 51 However, with both alumina and zirconia the etching reaction may be different. For example, alumina consists mainly of Al₂O₃ and zirconia mainly of ZrO₂ (Table 2-1). Because of these rather homogeneous compositions, no preferential etching may occur, and as a consequence, the contribution from micromechanical retention may be significantly reduced when compared to the other two ceramics. These results may be explained by assuming that such a difference exists between HC and IP ceramics on one hand and the PC and CZ ceramics on the other hand.

When it comes to alumina and zirconia, both are significantly tougher than the other two ceramics. Comparing zirconia with alumina reveals that zirconia-based material is the toughest of all the investigated ceramics (Figure 1-1). The high fracture toughness of zirconia is understood by examining the structure of zirconia. Zirconia may be present in a number of crystal phases, depending on the presence of small amounts of components such as calcia, magnesia, yttria (Y₂O₃), or ceria. A tetragonal precipitate of ZrO₂ is achieved by Y₂O₃ additions, while an alternative approach is used to produce a different microstructure. For example, a very
fine powder (<0.03 μm) containing 2 to 3 mol % Y₂O₃ and 97 to 98 mole % ZrO₂ can be densified completely in the tetragonal phase field to yield a fine-grained microstructure consisting almost totally of tetragonal grains. Each grain in this material can transform to another phase near a crack tip to inhibit propagation of the crack. When that happens, the ZrO₂ goes through a martensitic phase transformation from the tetragonal-to-monoclinic crystal form, causing the material to expand in front of the crack tip and induce a compressive stress in that region that hinders the crack to propagate.

Partly stabilized zirconia contains yttria, and its tetragonal zirconia phase is stabilized at room temperature. Under such conditions, etching will not produce a surface capable of forming a significant level of micromechanical retention, explaining why the bond strength of CZ treated with 9.6% hydrofluoric acid were lower within the same groups. From this observation, it can be determined that the 9.6% of hydrofluoric acid does not have a selective phase or is not strong enough to etch the zirconia-based ceramics. Hydrofluoric acid may just be an ineffective etchant on this type of ceramic.

Besides etching a ceramic surface, such a surface can also be sandblasted. By mixing air under pressure with a hard particulate abrasive, the ceramic material can be abraded or worn away from the surface of the ceramic. By using such an approach, a rough ceramic surface with a frosted appearance is produced during sandblasting. The longer time the abrasive stream targets the surface of the ceramic and the larger the size of the abrasive particulates, the more material will be removed. In this study, airborne Al₂O₃ particles, 50 μm in diameter, were used for five seconds with a pressure of two bars. By sandblasting the HC and IP ceramics, it is known that Al₂O₃ particles produce a uniform peeling appearance of the feldspathic ceramics or increase the number of pits per unit area of the IPS Empress ceramic. Such a treatment increases the
mechanical retention and the bond strength.\textsuperscript{54} Regarding the HC ceramic, there was no significant difference ($P > 0.05$) between sandblasted or acid etching. On that ceramic, it did not seem to matter which one of the resinous adhesives was used. IP showed almost the same results except that SXP resulted in the ($P < 0.05$) highest bond strength significantly when used on the etched IP surfaces.

For the PC and CZ ceramics, the shear bond strength values of the sandblasted surface were higher than the etched or finished surface treatments, which supports the hypothesis that air abrasion is the optimal surface treatment to roughen alumina and zirconia ceramic. This result was similar to Madani’s in 2000;\textsuperscript{55} however, these values were lower when compared to HC or IP ceramics. This difference may be related to the $\text{Al}_2\text{O}_3$ particles used during sandblasting having a higher fracture toughness than the HC or IP ceramics but having a similar fracture toughness as PC and lower than CZ. As a consequence, the lowest level of abrasion may have occurred on CZ followed by PC, while the highest abrasion occurred on HC and IP ceramics. In other words, the composition of the ceramic materials plays an important role when it comes to surface roughening ability.

**Adhesive**

The shear bond strength depends on the ceramic composition and the surface treatment of the ceramic. The silane enhances the wetting ability of the ceramics. When silane is applied to a ceramic surface and then dried, a condensation reaction occurs that results in a covalent bond formation with the glass surface. The silane-treated ceramic with its methacrylate groups present in the silane creates a bond with the methacrylate groups in the composite resin when the composite is cured (Figure 1-2).\textsuperscript{24}

CE is a single-component adhesive primer containing both silane and a phosphate monomer, 10-Methacryloyloxydecyl dihydrogen phosphate (MDP). When placed on a basic
surface, this phosphate monomer acts as an acid, loosens protons, and forms a negative backbone structure that reacts with positive charge sites on the ceramic surface. The silane also forms bonds to the ceramic surface. According to the manufacturer, phosphate monomer will bond directly to metal, alumina, or zirconia oxides, and as well as a silane coupler to bond with SiO2 based ceramics. MDP provides a long term-term stable bond to alumina and zirconia-based ceramics.\textsuperscript{48, 56} Regarding SXP, the surface treatment consisted of first a separate silane coating and then a coating with XP Bond. XP Bond contains dipentaerytritolpentacrylate-phosphoric-acid-monomer (PENTA-P) among other monomer systems, which reacts in the same way as MDP and bonds to positive charge sites present on a ceramic surface. However, Raffaelli (2004) concluded in his study that XP Bond was outperformed when compared to the control group.\textsuperscript{57} A possible explanation why Raffaelli (2004) did not succeed as well with XP Bond could simply be that he did not combine it with silane.

Considering these aspects, both CE and SXP can be described as both silane and acidic monomer treatments. Thus, both the silane and the acidic monomer will bond to ceramic surfaces and contribute to their good bonding abilities. Considering that SO does not contain any acidic monomer like MDP or PENTA-P, it seems reasonable to suggest that the higher bond strength values of CE and SXP are due to the presence of the acidic monomer components.

**Modes of Failure**

The clinical failure of all-ceramic restorations is very often associated with their brittleness and low fracture toughness. Recent studies show that the zirconia-based core material has the highest fracture toughness\textsuperscript{44}—twice that of alumina cores,\textsuperscript{45} which in turn is tougher than pressed leucite and feldspathic ceramics (Figure 1-1). Fracture toughness is an important material property and represents the ability of the material to resist brittle failure. The lower the fracture
toughness, the lower the clinical reliability of the ceramic restoration. The higher the fracture toughness, the better the material deflects a crack and distributes its energy.\textsuperscript{58}

The dominating failure mode occurred in the adhesive. Because of a lack of information (no SEM evaluation or microanalysis of surface compositions), researchers do not know whether failures occurred at the ceramic-adhesive interface, within the adhesive, or at the adhesive-composite interface. However, visual inspections indicate that most failures occurred within the adhesive film or at the adhesive-composite interface. The conclusion is based on the impression that a resin film was left on the ceramic surfaces after testing.

In addition to the adhesive failures, the HC and IP ceramics showed some cohesive failures occurring in the ceramic. These failures are attributed to high resin-ceramic bond strength values and sufficiently high stress levels transferred to the ceramic surface. In such situations, cracks that started at the interface could be diverted into the ceramic surface and result in a cohesive failure of the ceramic region in contact with the bonded composite surface. Such cohesive failures were seen in HC and IP, especially after surface etching, while no such failure was seen in any of the CZ or PC specimens.

The goal of this study was to determine the best method of repairing chipped ceramic veneering. Based on the results, the first recommendation, if possible, is that the clinician identifies the ceramic that has been used. If the chipped veneer is a FELD crown, the clinician should sandblast it and bond it with CE. In the case of an IP, the crown should be acid-etched and bonded with SXP. With CZ and PC, however, the ceramic should be sandblasted and bonded with SXP, as it preformed best in this study. If the ceramic restoration cannot be determined, repair of the chipped ceramic should utilize a universal system that works for all ceramics. Based on this study, the recommendation is to sandblast the ceramic surface and then use CE as the
bonding agent, as this method proved effective on each ceramic system tested. An interesting observation was that CZ had a higher shear strength value than the PC ceramic, when sandblasted with Al₂O₃. Since the fracture toughness of ZrO₂ is higher than the Al₂O₃, one would expect that sandblasting with Al₂O₃ should have less effect on ZrO₂ than on Al₂O₃. The reason would simply be that the Al₂O₃ particles rather than the zirconia surface should break during the impact. As a consequence, the zirconia surface should be less rough than the alumina surface after sandblasting and result in rather a lower than a higher bond strength than the alumina surface. However, the opposite was observed justifying future studies dealing with the impact of different sandblasting parameters.
Figure 3-1. Shear bond strength of HeraCeram

Figure 3-2. Shear bond strength of IPS Empress
Figure 3-3. Shear bond strength of the Cercon Zirconia

Figure 3-4. Shear bond strength of Procera Crown Alumina
Table 3-1. Feldspathic ceramic T-test comparison and significant differences of different surface treatments and adhesive bond materials.

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Table 3-2. IPS Empress ceramic T-test comparison and significant differences of different surface treatments and adhesive bond materials.

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Table 3-3. Procera ceramic T-test comparison and significant differences of different surface treatments and adhesive bond materials.

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Table 3-4. Cercon ceramic T-test comparison and significant differences of different surface treatments and adhesive bond materials.

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Figure 3-5. Type of HeraCeram ceramic adhesive failures of acid etched surface treatment.

Figure 3-6. Type of HeraCeram ceramic adhesive failures of sandblast surface treatment.
Figure 3-7. Type of IPS Empress ceramic adhesive failures of acid etched surface treatment.

Figure 3-8. Type of IPS Empress ceramic adhesive failures of acid etched surface treatment.

Figure 3-9. Failure of IPS Empress ceramic (IP) which had been etched before bonding (left). No such failures occurred in any of the Cercon Zerconia specimens (CZ), independent of surface treatment (right).
CHAPTER 4
SUMMARY AND CONCLUSIONS

The four formulated hypotheses showed to be difficult to reject or strongly support because of the interactions that occurred between ceramics, surface treatments and adhesives. The first hypothesis suggesting that hydrofluoric acid treatment would work best on feldspathic and pressed ceramics, was only supported for the pressed ceramic. The second hypothesis suggesting that air abrasion would provide better surface roughening on zirconia and alumina and therefore provide better bonding on these two ceramics than hydrofluoric acid was indirectly supported by the strength measurement and needs to be supplemented by surface roughness measurements. The third hypothesis suggesting that similarities between the silica structure and the silane molecules, should result in better bonding between the silane and feldspathic and pressed ceramics than with the Al₂O₃ and ZrO₂ ceramics was supported. The fourth hypothesis, suggesting that silane treatment is just a supplement to an etched or sandblasted surface was partly supported because in general the best adhesion occurred to surfaces with micromechanical retention ability. However, to prove this hypothesis, unsilanated specimens should have been included in the experimental design.

The key objective with this project was to determine how a clinician should repair a chipped ceramic restoration. The results revealed that different ceramics require different repair approaches and repair systems in order to perform best. The results suggest:

a) feldspatic ceramics performs equally well if sandblasted or etched.

b) IPS Empress performs best if etched with 9.6% hydrofluoric acid.

c) zirconia and alumina materials such as CER and PROC are best repaired by use of sandblasting.

d) Of the different adhesives, SXP and CE performed best on feldspathic, alumina and zirconia based ceramics, even though a more traditional treatment with SO performs equally well when used on feldspathic and pressed.
e) An important observation was that for sandblasted IPS Empress, the CE adhesive performed better than SXP. IPS Empress performed best after hydrofluoric acid treatment and when CE was used as adhesive.

In a situation, when the dentist does not know what kind of ceramics was originally used, sandblasting and use of CE will most likely give the best result.
LIST OF REFERENCES


BIOGRAPHICAL SKETCH

I was born in 1974 and grew up in Kuwait City, Kuwait. I graduated from Bayan High School in Kuwait City in 1992. In 1993, I enrolled at the University of Missouri-Kansas City to pursue a degree in biology of arts and science. In 1996, I was accepted at the University of Missouri-Kansas City School of Dentistry. In 1997, I married my beautiful wife, Dalal. In 2001, I graduated from the University of Missouri-Kansas City School of Dentistry with a Doctor of Dental Surgery degree. I have three children, two girls and a boy. I returned home to Kuwait and worked for the public health system of Kuwait from 2001 to 2005, before deciding to pursue a dental specialty degree in the United States. I enrolled in the graduate prosthodontics program at the University of Florida. After graduation with a Master of Science in dental sciences with specialization in prosthodontics from the University of Florida in July 2008, I will be enrolled in the same program as Implant Fellow for one year. On completion of the implant fellowship position, my wife and I plan to return to Kuwait and work for the public health system for the betterment of the dental service of our beloved country. This will be the beginning of the rest of our lives.