Flexural properties of leucite and lithium disilicate ceramic materials after repeated firings

Rifat Gozneli a*, Ender Kazazoglu b, Yasemin Ozkan c

a Department of Prosthodontics, Faculty of Dentistry, Marmara University, Nisantasi, Istanbul, Turkey
b Department of Prosthodontics, Faculty of Dentistry, Yeditepe University, Kadikoy, Istanbul, Turkey
c Department of Prosthodontics, Faculty of Dentistry, Marmara University, Nisantasi, Istanbul, Turkey

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Abstract Background/purpose: Pressable all-ceramic materials are widely used in dentistry. Determining the effect of repeated firing on flexural strength will help to improve these materials so that they can remain resistant to fracture in restorative work. The aim of this study was to determine the change in the flexural strength of pressable all-ceramic materials after repeated firings, which may be unavoidable when color and shape corrections are necessary for use in dental restorations.

Materials and methods: Forty disc specimens (15.5 mm x 2.1 mm) were prepared for each of four pressable ceramic materials (Empress 2, Finesse, Cergo, and Evopress) according to the manufacturers’ instructions. Each group of specimens was tested for biaxial flexural strength (piston on three balls test) after the first (n = 10), third (n = 10), fifth (n = 10), and seventh (n = 10) firing periods. The data were analyzed using one-way analysis of variance. The Tukey multiple comparison test was then used to compare the strength of the different materials, and the Newman–Keuls multiple comparison test was used to compare the strength at each firing interval measured (α = 0.05).

Results: The strengths of all of the pressable ceramic materials were decreased by repeated firings, especially after the seventh firing period, the only case in which the decrease was statistically significant. The flexural strength of the leucite-reinforced ceramic (Cergo) after the seventh firing was significantly lower than after the first firing (P = 0.04). The other materials were not significantly affected by repeated firings (P > 0.05).
Conclusion: The number of firings does not appear to significantly affect the flexural strength of pressable all-ceramic materials.

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Introduction

Ceramic materials have been used in dentistry for many years because of their esthetics and biocompatibility. However, their brittleness may cause failure of the restoration. Dental ceramics have been applied to metal substructures to produce porcelain-fused-to-metal fixed partial dentures to strengthen the ceramic structure. Subsequently, there have been numerous studies and reports on the effects of temperature or firing techniques on ceramic systems.

Stannard et al. and Hammad and Stein studied the effects of multiple firing on the shear bond strength of porcelain-fused-to-metal systems. Stannard et al. found that repeated firings of up to nine cycles did not significantly affect the bond strength between metal and porcelain. However, Hammad and Stein found that the increase in firing temperature increased the bond strength, while repeated firings tended to lower the bond strength of the metal substructure and porcelain, but not significantly.

Cattell et al. and Uctasli and Wilson studied the effects of different firing techniques on the flexural strength of pressable leucite-reinforced glass ceramics. No significant flexural strength changes were found between groups. Oh et al. studied the flexural strength changes of lithium disilicate reinforced Empress 2 all-ceramic material after different firing conditions. They found that the flexural strength values decreased slightly after the seventh firing, but this decrease was not statistically significant.

The challenge for most manufacturers has been the production of a ceramic material with sufficient strength and translucency. Strength is an important mechanical property that determines the performance of brittle materials. There are different testing methods available to assess the flexural strength of ceramic materials: the three-point bending test, the four-point bending test, the nondestructive test method, and the biaxial flexural strength test. The number of firings does not appear to significantly affect the flexural strength of pressable all-ceramic materials.

Several types of all-ceramic systems are available on the market. The most commonly used systems can be classified according to the laboratory processing procedure (pressable, slip-casting, milling, or sintering) and the chemical composition (feldspar: high leucite and low leucite; glass ceramic: mica, leucite, and lithium disilicate; core-reinforced: alumina, spinel injection molded, magnesia, and zirconia). Pressable glass ceramics are one of the most popular dental restorative systems due to several factors: ease of fabrication, occlusal accuracy, better marginal integrity, translucency, good mechanical properties, net-shaped forming by pressing, and decreased porosity.

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Several studies have investigated the effects of temperature or firing conditions on various dental porcelain systems. Claus reported that the firing cycle, temperature, rate of temperature increase, holding time, and cooling time all affect the distribution of the sintering, glass, and crystal phases in the microstructure of the porcelain.

<table>
<thead>
<tr>
<th>Materials</th>
<th>Type</th>
<th>Batch number</th>
<th>Manufacturer</th>
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<tbody>
<tr>
<td>Empress 2</td>
<td>Lithium disilicate pressable all-ceramic</td>
<td>S61194</td>
<td>Ivoclar Vivadent, Schaan, Liechtenstein</td>
</tr>
<tr>
<td>Finesse</td>
<td>Leucite-reinforced pressable all-ceramic</td>
<td>311200</td>
<td>Ceramco, Burlington, NJ, USA</td>
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<tr>
<td>Cergo</td>
<td>Leucite-reinforced pressable all-ceramic</td>
<td>0032/2</td>
<td>Degussa Dental, Hanau, Germany</td>
</tr>
<tr>
<td>Evopress</td>
<td>Leucite-reinforced pressable all-ceramic</td>
<td>41001</td>
<td>Wegold Edelmetalle, Wendelstein, Germany</td>
</tr>
</tbody>
</table>

Materials and methods

Specimen preparation

The all-ceramic materials selected for this study are listed in Table 1. Lithium disilicate ceramic (Empress 2; Ivoclar Vivadent, Schaan, Liechtenstein) and leucite-reinforced pressable all-ceramic (Finesse; Ceramco, Burlington, NJ, USA) were used. The materials were processed according to the manufacturer’s instructions. The specimens were then sintered at different temperatures and held at each temperature for a specified period. The specimens were then cooled at a specified rate. The flexural strength of the specimens was determined using a three-point bending test.
Vivadent, Schaan, Liechtenstein) and leucite-reinforced materials [Finesse (Ceramco, Burlington, NJ, USA), Cergo (Degussa Dental, Dusseldorf, Germany), and Evopress (Wegold Edelmetalle, Wendelstein, Germany)] were tested. Forty disc specimens were prepared for each material according to the manufacturers’ instructions.

Before pressing, prefabricated wax discs (Ivoclar Vivadent) of 15.5 mm radius and 2.1 mm thickness were sprued (Fig. 1) and invested by using each material’s own investment material. Each disc was then pressed according to the respective manufacturer’s pressing program. After pressing, the investment molds were taken from the furnace and allowed to air-cool. The investment material was then removed from the discs using an airborne particle abrasion unit (Toptec-Bego, Bremen, Germany) with 50-μm glass beads at a pressure of 4 to 2 bars (Fig. 2). The pressure level was decreased when closer to the ceramic material’s surface. The sprues were separated from the discs by use of a diamond disc bur (Horico, Berlin, Germany). Both surfaces of the specimens were serially wet-ground to the desired dimensions with 220-, 320-, 500-, 600-, and 800-grade silicon carbide papers mounted on a surface grinder and polisher machine (MetaServ Grinder-Polisher; Buehler UK, Coventry, UK). The disc specimens were fabricated with a 15.5-mm radius and 2-mm thickness, as indicated in ISO 6872 for the biaxial flexure test (piston on three balls) for pressed ceramics.28 Finally, the specimens were cleaned and washed under water.

Repeated firing

After surface finishing and polishing, each specimen was placed in its own furnace for the first firing program. This first firing served to release the stresses associated with the grinding and polishing procedures recommended by the manufacturers. Ten specimens from each material group were taken and designated 1.FG (1 Times Fired Group). The remaining specimens were refired a second time and then a third time. Ten specimens from each group of these remaining discs were designated 3.FG (3 Times Fired Group). The first three firings can be considered to be the minimum number of firings in both the staining and layering techniques, after pressing and finishing the ceramic restoration.

After two more firing periods, 10 specimens of each material formed the 5.FG (5 Times Fired Group). Finally, the remaining 10 specimens of each material were refired another two times and designated 7.FG (7 Times Fired Group). The firing program values were changed after each firing period according to each manufacturer’s instructions (Tables 2–5).

Biaxial flexure testing

The piston on three balls test was used to determine the biaxial flexure strength of the 160 discs after their first,

<table>
<thead>
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<th>Table 2</th>
<th>Firing program for Empress 2.</th>
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<td>Firing periods</td>
<td>Initial temperature (°C)</td>
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<tr>
<td>1</td>
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<tr>
<td>2</td>
<td>403</td>
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<td>403</td>
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<td>6</td>
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<td>7</td>
<td>403</td>
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</table>
third, fifth, and seventh firings. The test was performed with a universal testing machine (Testometric Micro 500; Testometric Company Ltd., Rochdale, Lancashire, UK) at a cross-head speed of 1 mm/min until fracture occurred. The disc specimens were supported on three steel balls (3.4-mm diameter) positioned 120° apart on a circle (11-mm radius). The force was applied to the center of the specimen with a flat surface loading piston of 1.4-mm radius, as recommended in ISO 6872 (Fig. 3). The recorded fracture load (in N) was then inserted into the following equation to give the flexural strength value (in MPa)28:

\[ S = -0.2387 \frac{P(X - Y)}{d^2} \]

where \( S \) is the maximum tensile stress (in MPa), \( P \) is the total load-causing fracture (in N), and \( d \) is the specimen thickness at the fracture origin. \( X \) and \( Y \) were determined as follows28:

\[ X = (1 + \nu) \ln \left( \frac{r_2}{r_3} \right)^2 + \frac{(1 - \nu)}{2} \left( \frac{r_2}{r_3} \right)^2 \]

\[ Y = (1 + \nu) \left[ 1 + \ln \left( \frac{r_1}{r_3} \right)^2 \right] + (1 - \nu) \left( \frac{r_1}{r_3} \right)^2 \]

where \( \nu \) is Poisson’s ratio, \( r_1 \) is the radius of the support circle (in mm), \( r_2 \) is the radius of the loaded area or the tip of the piston (in mm), and \( r_3 \) is the radius of the specimen (in mm) (Fig. 4). A value of 0.24 was assumed for the Poisson’s ratio of Empress 2,7 and one of 0.25 for the other materials.6

The statistical analysis in this study was performed using the GraphPad Prism V3 packet program (GraphPad Software Inc., La Jolla, CA, USA). In addition to calculation of the mean value and the standard deviation, a one-way analysis of variance was used to compare the measurements for the four material groups. To compare the strength values of the materials, a Tukey multiple comparison test was used. The Newman–Keuls multiple comparison test was used to compare the values after each firing period (\( x = 0.05 \)).

### Results

The biaxial flexural strength values of the four all-ceramic materials after each firing period are listed in Table 6. There were statistically significant differences between the flexural strength values of the materials containing lithium disilicate and leucite at all firing periods (\( P < 0.0001 \)). Of the materials tested in the study, the lithium disilicate ceramic (Empress 2) had the highest value for flexural strength (250.04 ± 23.22 MPa), more than twice that of the leucite ceramic materials.

The Tukey multiple comparison test showed that, within the leucite ceramic materials (Finesse, Cergo, and Evopress), the flexural strength values were not significantly different from each other for most of the firing periods. Only Finesse had a higher flexural strength than Evopress at the seventh firing period (\( P < 0.01 \)).

Repeated firing was found to be associated with a slight decrease in the flexural strength values of all the tested materials, especially after the seventh firing period. However, the results were not statistically significant for all materials (Fig. 5). The flexural strength values after repeated firing were found to be statistically significant only for Cergo pressable ceramic, with a significant change being detected between the first and seventh firing periods for Cergo (first, 117.27 ± 14.18 MPa; seventh, 104.71 ± 10.26 MPa; \( P = 0.04 \)).
Discussion

This in vitro study measured the changes in biaxial flexural strength of pressable all-ceramic materials after repeated firing. Within the limitations of this study, the results do not support our hypothesis regarding the effect of repeated firing on the biaxial flexural strength of pressable all-ceramic materials. The strength values for all the materials were decreased by repeated firing at the seventh firing period, but the difference was not statistically significant.

Ceramics have many uses in restorative dentistry because they have so many esthetic and physical advantages. In particular, all-ceramic systems are preferred because of their light-transmitting features. In this study, the most commonly used pressable all-ceramic materials were selected for testing.

Although the relationship between the mechanical properties of a ceramic material and its clinical performance may be influenced by many variables, strength has often been the first parameter investigated to determine the clinical potential and limits of a dental ceramic. There are several different testing methods available for assessing the flexural strength of the ceramic materials. The three-point bending test is considered to be the standard for strength-testing of dental ceramics. However, the disadvantage of this test is its inherent sensitivity to flaws and defects near the edges of the specimens. This problem is eliminated by the use of the biaxial flexural strength test, which is not affected by defects produced under stress.

The optimum strength of ceramics is related to the fabrication procedure and is achieved by minimization of flaws in the content. In addition, Claus reported that the firing cycle and temperature affect the distribution of the sintering, glass, and crystal phases in the microstructure of the porcelain, which may affect the optimum strength of all-ceramic restorations. Baharav et al investigated the effect of glazing time on the fracture toughness of alumina porcelain, and Lenz et al examined the effect of different firing temperatures on the flexural strength of dentin porcelain. Many researchers have also studied the effect of the firing procedure on the strength of porcelain. However, more research is needed into the effects of repeated firing, especially for today’s most popular all-ceramic systems.

In studies on the repeated firing of ceramics, firing periods were limited to between one and nine times. The first firing serves to eliminate microcracks and release the stresses associated with the grinding and polishing procedures, as recommended by the manufacturers. The second and third firings are considered to be necessary steps for producing the restoration using the staining or layering technique, prior to installation in the mouth.

<table>
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<th>Table 5 Firing program for Evopress.</th>
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<td>Evopress</td>
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<tr>
<td>Firing periods</td>
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<td>1</td>
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<tr>
<td>2</td>
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Figure 3 Biaxial flexure testing.

Figure 4 Diagram of the piston on three balls test. F = force; r = radius.
fourth and subsequent firings are necessary when shape and color corrections are required. It is assumed that, after the third firing, an all-ceramic restoration is ready for installation in the mouth by the dentist. The final four firings, after the third firing, were assumed to be necessary only if the dentist needed further shape and color corrections for the ceramic restoration.

In most of the studies, the wax specimens were produced with handmade molds. However, in this study, prefabricated and presprued wax discs (Ivoclar Vivadent) were used to eliminate dimensional errors between specimens. The dimensions of the specimens conformed to ISO 6872.28 Because of the prefabricated wax discs, small values were obtained for standard deviation.

It is known that leucite ceramics exhibit an increase in leucite content after repeated firing,29,30 and the size of lithium disilicate crystals in ceramics containing lithium disilicate has been found to increase after repressing.31 Although these kinds of changes in the microstructure of ceramics containing leucite and lithium disilicate have been reported, in this study the flexural strength of these ceramics was not affected by repeated firing. Thus, it may be considered that the changes in the microstructure of a leucite and lithium disilicate ceramic do not always affect its flexural strength values. Further investigation of pressable ceramics should investigate the effects of repeated firing on the bond between the core and layering materials.

Within the limitations of this study, the following conclusions can be drawn:

1. The lithium disilicate ceramic (Empress 2) had the highest flexural strength at all firing periods, more than twice that of the other materials.
2. The strength values of all the pressable ceramic materials’ were decreased by repeated firing, especially after the seventh firing period, although the results were found to be statistically significant for only one of the materials.
3. The flexural strength values after repeated firing were found to be statistically significant only for Cergo pressable ceramic, and only between the first and seventh firing periods.

Conflicts of interest

The authors have no conflicts of interest relevant to this article.

Acknowledgments

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