Heat-Pressed Ceramics: Technology and Strength

The flexural strength of a new heat-pressed ceramic material (IPS-Empress) was measured before and after pressing and/or simulated firing treatments (e.g., veneering, surface coloring, glazing). Heat pressing the material significantly improved its flexure strength whereas heat treating the material alone did not. Additional firings (heat treatments) after heat pressing further increased material strength. The final strength values ranged between 160 and 180 MPa and were comparable to some other all-ceramic systems. No clinical implications were drawn from these data. *Int J Prosthodont* 1992;5:9-16.

Recently, several new all-ceramic crown systems that offer good esthetics and simplify fabrication have been introduced. With these systems it is possible to fabricate single crowns, inlays, and veneers. On vital teeth the advantages of using a translucent ceramic material with characteristics similar to those of natural enamel are particularly apparent. Castable glass-ceramic and refractory die techniques have been judged to meet these requirements. The disadvantage of the refractory die system is that the particles are sintered together, resulting in microporosities and inhomogeneities between the ceramic particles. It is well known that these microporosities can initiate crack propagation, leading to early failure of such all-ceramic restorations.6 Many castable glass-ceramic systems have recently been described (Dicor,7 Cera Pearl,4,5 and Olympus Castable Ceramics6) in which the porosities can be reduced to a minimum by the casting technique. However, the casting process is followed by a ceramming procedure (controlled crystallization using an appropriate heat treatment), resulting in additional ceramic shrinkage.7 To overcome this disadvantage, a heat-press technique (IPS-Empress) was developed in 1983 by the Department of Fixed and Removable Prosthodontics and Dental Materials at the University of Zurich. Since 1986 the development has proceeded in conjunction with a dental company (Ivoclar, Schaan, Liechtenstein).

A patented heat-press technique was first described in 1936 for the construction of ceramic complete dentures.8 However, to the authors' knowledge, a commercially available furnace was never developed. In 1969, Droge9 described a ceramic press technique based on the hot-press resin technique. In his work he was using two-part flasks. Instead of stone, the wax crown was invested in a refractory die material. After opening the two halves of the flask and removing the wax with boiling water, the cavity was filled with ceramic powder and pressed together in the hot stage. Improving Droge's technique, McPhee10 was able to produce complete-coverage metal-ceramic...
restorations that accurately duplicated occlusal surfaces. The glass-ceramic ingot used in this paper, called IPS-Empress, is precerammed and preco-colored. This system is designed for the fabrication of single units, inlays, and veneers. Natural teeth and IPS-Empress crowns appear to be similar, and therefore good esthetic results can be achieved with good marginal integrity. For the inlay technique, the IPS-Empress system offers easy handling and good colormatching because of the similarity between the translucency of the base material and the tooth structure.

The first part of this paper describes the material, its development, and the fabrication technique; the second part discusses the material and its strength. Specifically, the purpose was to determine whether pressing and/or heat treatment (simulated firing) affect the flexural strength of the ceramic.

Materials and Fabrication Procedures

The glass-ceramic material developed for this technique is basically a feldspathic porcelain having the following composition in wt%: 63 SiO₂, 17.7 Al₂O₃, 11.2 K₂O, 4.6 Na₂O, 0.6 B₂O₃, 0.4 CeO₂, 1.6 CaO, 0.7 BaO, 0.2 TiO₂. The crystalline part of the ceramic consists of leucite crystals.

A special furnace (Empress EP 500) was designed for this system. The pressing furnace contains an enlarged heat dome, a pneumatic pressure system, a reducing valve, and a manometer to control the pressure; an inductive displacement transducer is mounted on top of the furnace and is connected to the pneumatic plunger. The crown or inlay was waxed and placed on a specially designed cylindrical crucible former and invested using a phosphate-bonded investment. The mold was heated in a burnout furnace to 850°C. The cylindrical opening into the mold was filled with a ceramic ingot and an Al₂O₃ pushing rod. The assembly was then placed into the preheated furnace. The automatic press furnace makes it possible to change the following parameters:

1. The rate of temperature increase can be varied from 5°C to 80°C/min.
2. The furnace can be heated to 1,200°C.
3. The holding time at the final temperature can be varied from 0 to 60 minutes.
4. If the pneumatic plunger does not continue to move more than 0.3 mm/min, the pressure maintenance time will be activated. A pressure maintenance time of 1 to 4 minutes is necessary depending on the thickness of the cavity that has to be filled; the time can be varied from 1 to 20 minutes.
5. The press procedure is performed in a vacuum, and the beginning and ending points for the vacuum application can be programmed. When the start button is pushed, the furnace heats up automatically to the programmed press temperature (1,150°C). After a 20-minute holding time.
at this temperature the press procedure was activated and the then-plastic glass-ceramic material was pressed (0.3 to 0.4 MPa) into the mold. The mold was filled with the glass-ceramic material and the furnace stopped automatically. The ceramic restorations were devested and prepared for further treatments (Fig 5).

Fig 3 Ceramic ingots are preshaded and precerammed. For the inlay technique, translucent material is used.

Fig 4 After filling the cylindrical opening with an already preheated ceramic ingot and an $\text{Al}_2\text{O}_3$ pushing rod, the cast must be placed into the preheated Empress furnace. The aluminium oxide pushing rod is used to transfer the pressure to the ceramic material.

Fig 5 After the press procedure, the inlays are devested and prepared for further treatments.

Two methods may be used to obtain the desired shade.

1. Surface coloration: A strongly pigmented surface color is applied until the desired color is established. The color pigments are covered with a surface glaze that consists of the same components as the base material. The surface
Fig 6. The occlusal surface and the inner surface can be covered with a thin layer of surface stains. The occlusal surface will be covered with a glaze.

Figs 7a and 7b. Inlays can be made more simply and have good marginal integrity when placed.

Figs 8a and 8b. Examples of the surface staining technique (left) and the veneer technique (right). A complete waxing must be made by both techniques. When using the veneer technique, the enamel part is cut back and the dentin structure is pressed. The base of the precolored ingot is adjusted to the Ivoclar and Vita shade guide. The final form is established using enamel porcelain. Using the surface staining technique with a strongly pigmented surface color, the crown can be characterized until the desired color is established. These color pigments will be covered in two additional firings with a glazing material having the same components as the base material.
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Fig 9 Cross section (18X) of the surface colored crown shown in Fig 8b. The surface colors are covered with a glaze compound in a thickness of about 50 μm.

Fig 10 Cross section (18X) of the veneered crown shown in Fig 8b. Many metal oxides that are responsible for the color of the base material are visible.

glaze is applied in a thickness of approximately 50 to 60 μm (Figs 6 through 10).

2. Veneer technique: A complete-contour wax pattern is made and then a portion is removed where enamel translucency is desired. The remaining dentin structure is then invested and heat pressed. After devesting and sprue removal, the dentine structure can be characterized using surface colors. Finally, enamel porcelain is applied to complete the desired form.

**Flexural Strength**

The second part of the study determined the influence of pressing and/or heat treatment (simulated firing) on the flexure strength of the glass-ceramic material. The IPS-Empress material (63-PK/ST II, Ivoclar) was used to make (2 X 4 X 14)-mm flexure strength specimens. Eight groups (n = 15) of bars were prepared according to the treatments listed in Tables 1 and 2. For groups 1 through 3, the cylindrical ingots (10.5 mm in diameter and 7.5 mm in height) were not heat pressed but were machined in the as-received state (group 1) or after heat treatments (groups 2 and 3). A slow-speed diamond wire saw (Well, W Ebner, Switzerland) was used to machine the samples. The bar surfaces were ground flat with a rectangular cross section, using a metallurgical grinding machine (Knuth-Rotor, Struers, Denmark) with SiC discs (grit size 320).

For the remaining groups (4 through 8) the ingots were first heat pressed into bars of the required geometry. The bars were then heat treated (Tables 1 and 2) and ground to the final form. The detailed procedure was as follows: wax bars were made by injecting casting wax (S-U-Tauchwachs, Schuler Dental, Germany) into a silicone mold. Sprue formers (2 mm in diameter) were then attached to a specially designed crucible former and invested.

**Table 1** Pressing and Firing Conditions of Each Group

<table>
<thead>
<tr>
<th>Group no.</th>
<th>Pressing</th>
<th>Firing conditions</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>-</td>
<td>No firing (ingot)</td>
</tr>
<tr>
<td>2</td>
<td>-</td>
<td>Simulated press firing</td>
</tr>
<tr>
<td>3</td>
<td>-</td>
<td>Simulated press and veneer firing</td>
</tr>
<tr>
<td>4</td>
<td>+</td>
<td>Practical pressing</td>
</tr>
<tr>
<td>5</td>
<td>+</td>
<td>Simulated veneer firing</td>
</tr>
<tr>
<td>6</td>
<td>+</td>
<td>Simulated veneer and glaze firing</td>
</tr>
<tr>
<td>7</td>
<td>+</td>
<td>Simulated surface coloring and glaze firing</td>
</tr>
<tr>
<td>8</td>
<td>+</td>
<td>Simulated veneer, surface coloration, and glaze firing</td>
</tr>
</tbody>
</table>

**Table 2** Firing Conditions of the Empress System

<table>
<thead>
<tr>
<th>Firings</th>
<th>Temperature (°C)</th>
<th>Heating rate (°C/min)</th>
<th>Holding time (min)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Press firing*</td>
<td>850 - 1150</td>
<td>60</td>
<td>20</td>
</tr>
<tr>
<td>Veneer firing</td>
<td>400 - 920</td>
<td>60</td>
<td>2</td>
</tr>
<tr>
<td>Surface coloring</td>
<td>400 - 850</td>
<td>60</td>
<td>1</td>
</tr>
<tr>
<td>Glaze firing</td>
<td>400 - 870</td>
<td>60</td>
<td>1</td>
</tr>
<tr>
<td>Final glaze firing</td>
<td>400 - 890</td>
<td>60</td>
<td>1</td>
</tr>
</tbody>
</table>

All firings are under vacuum except final glaze firing. *Pressing after 20-minute holding time.
Table 3  Flexure Strength Results

<table>
<thead>
<tr>
<th>Group no.</th>
<th>Mean strength (MPa)</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>74 (10)</td>
</tr>
<tr>
<td>2</td>
<td>91 (11)</td>
</tr>
<tr>
<td>3</td>
<td>82 (11)</td>
</tr>
<tr>
<td>4</td>
<td>126 (15)</td>
</tr>
<tr>
<td>5</td>
<td>182 (26)</td>
</tr>
<tr>
<td>6</td>
<td>175 (32)</td>
</tr>
<tr>
<td>7</td>
<td>159 (28)</td>
</tr>
<tr>
<td>8</td>
<td>171 (29)</td>
</tr>
</tbody>
</table>

Standard deviations are in parentheses.

The following group-specific heat treatments were performed (Table 2):

Group 1  No treatment was used.
Group 2  The ingots were fired on a flat refractory tray for 30 minutes (Table 2) at the usual pressing temperature but without pressing (simulated press firing).
Group 3  After a simulated press firing, the ingots were placed on a flat refractory tray in a porcelain furnace (Programat P90, Ivoclar) and subjected to two simulated veneer firings.
Group 4  There was no heat treatment after pressing.
Group 5  The heat-pressed bars were subjected to two simulated veneer firing cycles.
Group 6  The pressed bars were subjected to the same heat treatment as those in group 5, with three simulated glaze firings and one simulated final glaze firing.
Group 7  The pressed specimens were subjected to three simulated surface coloration and glaze firings and to one simulated final glaze firing.
Group 8  The pressed bars were subjected to two simulated veneer firings, three surface coloration firings, three glaze firings, and one final glaze firing.

A three-point bending test was conducted using a universal testing machine (RM 50, Schenck-Trebel, Germany) at a cross-head speed of 0.5 mm/min. The data were analyzed using ANOVA. Scanning electron microscope (SEM) analyses (Model 1810, Amray) were done on etched (0.2% hydrofluoric acid/5 min) fracture surfaces to relate the revealed microstructure (leucite size, shape, and distribution) to the mechanical behavior of the ceramic material.

![Fig 11](image1.png)  Flexure strength comparison of Empress after simulated press and veneer firing with the ingot.

![Fig 12](image2.png)  Flexure strength comparison of Empress after pressing and simulated veneer firing with the ingot.
Results

The experimental strength results of the eight groups—means and standard deviations (SD) expressed in MPa—are listed in Table 3. Figure 11 shows the strength comparison among groups 1 through 3. A statistical evaluation (comparing groups 3 and 2, groups 3 and 1, and groups 2 and 1) indicates that heat treating the material through a simulated firing in the absence of pressing does not significantly ($P > .001$) affect its strength.

Figure 12 shows the strength comparison among groups 1, 4, and 5, where the influence of pressing is taken into account. The statistical evaluation (comparing groups 4 and 1, groups 4 and 5, and groups 1 and 5) indicates that heat pressing the material (group 4) does significantly ($P < .001$) increase its strength. Subsequent heat treatment (group 5) also significantly increases strength.

There was no significant difference in strength between the four heat-treatment techniques (groups 5 through 8) used after the material had been heat pressed (Fig 13).

Discussion

This study indicates that heat pressing is not only a method of shape forming, but it also has a positive influence on the strength of the ceramic material even though the applied pressure of 0.4 MPa is quite moderate. If pressing is additionally followed by firing, the strength of the ceramic significantly increased and reached a high mean value of 182 MPa (group 5). Consequently, the combination of heat pressing and heat treatment (simulated firing) produced the greatest strength.

Evaluation of the microstructure provided additional information. The SEM photograph (Fig 14) of an as-received Empress ingot shows the leucite crystals grouped in cluster forms and located in large areas of the glassy phase. In contrast, the heat-pressed material exhibited much better leucite dispersion (Fig 15). One possible explanation is that the clusters were separated during the forcing of the softened ingot through the small-diameter sprue. The crystals (average size, 1 $\mu$m) were consequently distributed individually and in a more homogeneous manner in the glassy phase. It is reasonable to suppose that the improved strength of the pressed ceramic material resulted from the better dispersion of the leucite crystals in the glass matrix.

The additional strength resulting from the simulated firing was reportedly caused by the different thermal expansion coefficients ($\alpha$) of glass and crys-
Leucite is a high-expansion material with a nonlinear dilation curve. If measured between 20°C and 300°C its value is 17 × 10⁻⁶/°K; in the temperature interval of 500°C to 625°C its value rises to 65 × 10⁻⁶/°K. Calculated between 20°C and 625°C, α has a value of 27 × 10⁻⁶/°K. The thermal expansion coefficient of glass is far lower (in order of 10 × 10⁻⁶/°K). It is known that, on cooling, the presence of a high-expansion coefficient crystal in a matrix of a lower-expansion coefficient glass generates tangential compressive stresses in the glass and radial stresses within the crystal. The compressive stresses have the effect of increasing the specimen's resistance to surface damage by opposing the formation of surface cracks.

All examined firing conditions (Table 2) ranging between 850°C and 920°C have the same beneficial effect on flexure strength material when color was previously pressed (Fig 13). The values range between 160 and 182 MPa and are close to the strength results of some other all-ceramic systems (e.g., 180 MPa for Hi-Ceram core material from Vita Zahnfabrik, Bad Sackingen, Germany) tested under the same conditions. However, no conclusion with respect to clinical implication can be drawn from these data.

Summary and Conclusions

This paper described a new technique of fabricating all-ceramic inlays and crowns using a heat-pressed glass-ceramic material (IPS-Empress). The effect of heat pressing and subsequent heat treatment upon the flexural strength of the material was studied and the following conclusions were made:

1. Heat pressing of the material as received from the manufacturer significantly improved its strength (from 74 to 126 MPa).
2. Subsequent heat treatments (simulated veneer firings, surface coloration firings, or glaze firings) further increased material strength to values ranging from 160 to 182 MPa.
3. Heat treatment in the absence of pressing had no effect on strength.

Acknowledgments

The authors would like to thank Dr Loganathan Vijayan, visiting from National Hospital of Singapore, for his advice and help with the manuscript. We are grateful as well for the support from staff members at the Department of Crown and Bridge Prosthodontics and Dental Materials, Dental Institute, University of Zurich. Namely, the assistance of Mr Olivier Loeffler and Mrs Renate Loeffler with materials testing and SEM work, respectively, was very much appreciated.

References