

# **Fracture Loads of All-Ceramic Crowns under Wet and Dry Fatigue Conditions**

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#### Keywords

Composite resin cement; resin-modified glass ionomer cement; ceramic system; luting agent.

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## Abstract

**Purpose:** The aim of this study was to test the hypothesis that fracture loads of fatigued dental ceramic crowns are affected by testing environment and luting cement.

**Materials and Methods:** One hundred and eighty crowns were prepared from bovine teeth using a lathe. Ceramic crowns were prepared from three types of ceramic systems: an alumina-infiltrated ceramic, a lithia-disilicate-based glass ceramic, and a leucite-reinforced ceramic. For each ceramic system, 30 crowns were cemented with a composite resin cement, and the remaining 30 with a resin-modified glass ionomer cement. For each ceramic system and cement, ten specimens were loaded to fracture without fatiguing. A second group (n = 10) was subjected to cyclic fatigue and fracture tested in a dry environment, and a third group (n = 10) was fatigued and fractured in distilled water. The results were statistically analyzed using one-way ANOVA and Tukey HSD test.

**Results:** The fracture loads of ceramic crowns decreased significantly after cyclic fatigue loading ( $p \le 0.05$ ); furthermore, fracture loads of crowns fatigued in a wet environment were statistically lower than those in a dry environment (p < 0.05). Crowns luted with a composite resin cement showed statistically greater fracture loads than those luted with a resin-modified glass ionomer cement ( $p \le 0.05$ ).

**Conclusions:** Fracture load of the three ceramic systems was found to be influenced by ceramic composition. Moreover, cement and fatigue condition influenced the fracture loads of the crown specimens evaluated in this study.

Ceramics as dental materials have desirable characteristics such as chemical stability, biocompatibility, high compressive strength, and a coefficient of thermal expansion similar to that of tooth structure.<sup>1</sup> In addition, dental ceramics have esthetic properties that simulate the appearance of natural dentition;<sup>2</sup> however, they are susceptible to fracture, which is a result of material characteristics and surface and bulk defects.<sup>3,4</sup>

Metal-supported ceramics were introduced to improve the clinical survival of the overall restoration;<sup>5</sup> however, metal core reduces the translucency and adversely affects the esthetics of the restoration. Advanced ceramics with various crystalline phases and processing techniques have been developed to achieve greater strength and toughness and to avoid the use of

metal core.<sup>6</sup> Examples of strengthened ceramics are In-Ceram Alumina (Vita Zahnfabrik, Seefeld, Germany), IPS Empress 2 (Ivoclar-Vivadent, Schaan, Liechtenstein), and Cergogold (Degussa Dental, Hanau, Germany).

In-Ceram Alumina is an aluminous ceramic with 82% volume alumina, infiltrated by glass,<sup>7</sup> and it is recommended for anterior and posterior full crowns, and anterior three-unit fixed partial dentures (FPDs). IPS Empress 2 is a multiphase glass ceramic with 60% volume of two crystal phases: lithia-disilicatebased ( $Li_2O$ ·SiO<sub>2</sub>) crystals as the main phase, and lithium orthophosphate crystals as the second phase.<sup>8</sup> This material is used to fabricate anterior and posterior full crowns, and anterior three-unit FPDs.<sup>9</sup> Cergogold is a glass-ceramic material containing leucite crystals,<sup>10</sup> and it is suggested for fabricating only single-unit crowns.

Despite the extensive use of reinforced ceramic systems, some factors, such as microdefects within the materials, improper design, high impact load, and fatigue, have been shown to cause intraoral ceramic fracture.<sup>11,12</sup> Fatigue fracture is a form of failure that occurs in structures subjected to dynamic and fluctuating stresses.<sup>13</sup> Under these conditions, it is possible for cracks to propagate at a stress level considerably lower than the strength of the dental ceramic. Catastrophic fracture results from a final load that exceeds the load-bearing ability of the remaining sound portion of the material.<sup>11</sup> Studying fatigue resistance of ceramic dental prostheses will provide a more detailed understanding of failure in clinical use. Furthermore, even though the results of in vitro studies cannot be entirely correlated with in vivo conditions, forces in the oral environment fluctuate.<sup>14</sup>

In addition to the fracture resistance of dental prostheses, luting materials are also important for the longevity of dental restorative materials. Another factor affecting the fatigue strength of dental ceramics is the environmental condition. Previous researchers have shown that the final fracture load of ceramics after fatigue depends on the environmental condition, that is, dry or wet.<sup>15,16</sup> To date, limited information is available on the fatigue of all-ceramic systems, especially for those using teeth substrate and different cement systems. Therefore, the aims of this study were to investigate the fatigue resistance of dental crown shapes fabricated using three ceramic systems (In-Ceram Alumina, IPS Empress 2, and Cergogold) in dry and wet environments and to study the effect of two cements, a composite resin cement, and a resin-modified glass ionomer cement, on the fatigue resistance of dental crown prostheses. The null hypotheses were: (1) the type of cement does not affect the fatigue strengths of the three ceramic systems; (2) the environmental conditions do not affect the fatigue strengths of the three ceramic systems; and (3) there is no statistically significant difference in fracture loads of the three ceramic materials with the same luting cement and the same testing conditions.

## **Materials and methods**

One hundred and eighty bovine mandibular incisors were collected and stored in a 10% formalin solution.<sup>17</sup> Calculus deposit and soft tissue were removed from the selected teeth with a scaler and cleaned with a rotational brush and nonfluoridated flour of pumice (Zircate Prophy Paste, Dentsply, Milford, DE). Mechanical retentions were made in the root parts of the teeth to ensure that during preparation, the teeth position would be stable. The teeth were embedded with autopolymerizing acrylic resin (Clássico, Produtos Odontológicos, Sao Paulo, Brazil) in polyvinyl chloride tubes (Tigre, Joinvile, Brazil), which were 25.4 mm in diameter and 30 mm in height. The teeth were placed upright with the long axes parallel to the height of the tube; the cementoenamel junctions (CEJ) were located 3 mm above the resin. The assembled specimens were attached to a lathe (Nardini-ND 250 BE, São Paulo, Brazil) with a grinding device and prepared under water spray. The final dimensions of the teeth specimens were  $7.0 \pm 0.5$  mm in height, 8.0 mm



Figure 1 Tooth preparation.

cervical diameter, 4.2 mm occlusal diameter (Fig 1). A 0.8 mm-deep shoulder finish line with a rounded internal line angle was prepared using a diamond instrument (No. 5850–018, Brasseler USA, Savannah, GA). The teeth were prepared with an 8° angle of convergence. All sharp angles were rounded, and all finish lines were located  $1.0 \pm 0.2$  mm above the CEJ. All teeth were measured after preparation using a precision electronic micrometer (Electronic Micrometer, LS Starrett, Athol, MA) with an accuracy of 0.002 mm.<sup>18</sup>

The 180 prepared teeth were divided into three groups (n = 60) as follows: In-Ceram Alumina, IPS Empress 2, Cergogold. A two-stage impression was made for each prepared tooth with a polyvinyl siloxane (PVS) impression material (Express, 3M ESPE, St. Paul, MN) using a custom-made impression tray fabricated with acrylic resin. Then, type IV gypsum (Fuji Rock, GC America, Alsip, IL) was poured to produce dies.

The dies were coated with one layer of die spacer (Spacelaquer Ducera Lay, Degussa Huls, Hanau, Germany) to approximately 1 mm above the finish line. For IPS Empress 2 and Cergogold, the dies were coated with lubricating oil (Die Lube, Dentaurum J.P. Winkelstroeter KG, Pforzheim, Germany), and 0.7-mm thick wax patterns were fabricated over the master dies using a wax dipping unit (Hotty, Renfert, Hilzingen, Germany). Following the preparation of the wax patterns, each pattern was sprued and invested in an investing ring. A two-stage burnout sequence was used: (1) heat at 5°C/min to 250°C, 30-minute hold; and (2) heat at 5°C/min to 850 °C, 1-hour hold. After the preheating stage, the investment cylinders were immediately transferred to the pressing furnace (EP500, Ivoclar AG). The pressing temperatures for Empress 2 core and Cergogold core ceramics were 920°C and 850°C, respectively. Following the pressing procedure, the investment cylinders were removed from the pressing furnace and cooled for 2 hours in a ventilated room. The cooled specimens were divested by grit blasting with 80 mm glass beads (Williams glass beads, Ivoclar North America, Amherst, NY). Before etching, the sprues were cut away, and excess sprue segments were removed by grinding

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from the specimen surfaces using water as a coolant. The core specimens were placed in one plastic bottle containing 20 ml of 1% hydrofluoric acid solution (Invex Liquid, Ivoclar AG), and these bottles were placed in an ultrasonic bath. After etching, the specimens were cleaned under running tap water for 10 seconds and then dried thoroughly. These procedures were performed by a certified dental technician.

For the In-Ceram Alumina, three layers of die spacer (Vita Zanfabrik) were applied on the stone die surface to approximately 1 mm above the finish line. Impressions were made using a PVS impression material (Express) with a plastic ring. These impressions were poured with In-Ceram special plaster using a liquid-to-power ratio of 0.23 ml/g to make refractory models. In-Ceram powder slip was prepared according to the manufacturer's instructions and was applied to the models. A sculpturing device was used to ensure a uniform core thickness.<sup>19</sup> After applying a stabilizer, the coping was fired on the plaster dies and infiltrated with glass. Excess glass was removed with a diamond bur. These procedures were conducted in an authorized laboratory by a certified technician. The final dimension of IPS Empress 2 and Cergogold copings were 0.7 mm and for In-Ceram it was 0.5 mm.

The veneer porcelains (D'Sign, Ivoclar for IPS Empress 2 core; Vitadur alpha, Vita Zahnfabrik for In-Ceram core; Duceragold, Degussa Dental for Cergogold core) were applied on to the core materials, which had been placed in a split brass mold to make a complete crown shape with a stratification porcelain thickness measuring 0.3 mm for IPS Empress 2, Cergogold, and In-Ceram specimens in the cervical region and increasing in thickness in accordance with the angle of convergence. Following veneer porcelain sintering, the final dimensions of the layered porcelain-coated specimens were 0.2 mm cervical, 1.0 mm in the pulp axial angle, and 1.5 mm in occlusal aspect.<sup>16</sup> The teeth were stored in distilled water at  $37^{\circ}$ C until the cementation process.

The crown shapes were cemented onto the teeth preparations. The 60 crowns in each ceramic system were divided in two groups (n = 30) with each group having a different cement system, that is, a composite resin cement (Variolink II, Ivoclar Vivadent) and a resin-modified glass ionomer cement (Rely X Luting, 3M ESPE Dental Products, St. Paul, MN).

#### **Application of cement**

Variolink II: Teeth surfaces were cleaned with a rotational brush and nonfluoridated flour of pumice (Zircate Prophy). The dentin was treated for 15 seconds with 35% phosphoric acid and rinsed for 10 seconds under running tap water. Excess water was removed with a cotton pellet, leaving a moist surface. Two consecutive coats of adhesive were then applied using a saturated brush tip. The ceramic surface was etched with 10% hydrofluoric acid (Ácido hidrofluorídrico, Dentsply Brazil, Petropolis, Brazil) for 1 minute [Cergogold and In-Ceram (hydrofluoric acid was used for In-Ceram for cleaning purposes only)] or for 20 seconds (IPS Empress 2), followed by rinsing for 1 minute. Specimens were then ultrasonically cleaned with distilled water for 10 minutes and dried with oil-free air. The silane agent Monobond S (Ivoclar Vivadent) was applied, and the surface was dried after 1 minute using compressed air. The composite resin cement was applied to the internal ceramic crown surface. A load of 454 g was applied while the excess cement was removed. The composite resin cement was light cured (XL3000, 3M ESPE Grafenau, Germany) for 40 seconds on each side (labial, lingual, mesial, and distal) of the crown resulting in 160 seconds of light polymerization for each crown with 500 mW/cm<sup>2</sup> light intensity. Ten minutes after the mixing began, specimens were immersed in distilled water at 37°C and stored until testing.

Rely X Luting: The procedures were the same as the aforementioned composite resin cement, but the dentin was simply cleaned and did not receive any adhesive application. The ceramic surfaces were cleaned in the same manner as described in the resin cement application procedures above; however, they did not receive the silane application.

#### **Fatigue resistance measurement**

The 30 crown specimens in each group were divided into three subgroups (n = 10 for each subgroup). The first subgroup was tested without fatigue loads after 24 hours of storage in distilled water at 37°C using an Instron universal testing machine (Instron 4411, Instron, Canton, MA). A preload of 20 N was applied to the center of the occlusal surface of the crown sample with a 4-mm-diameter stainless steel ball, followed by a crosshead speed of 1.0 mm/min until fracture occurred. In the other two subgroups, the crown specimens were submitted to a fatigue test of 60,000 cycles that consisted of cyclic loading between minimum and maximum loads of 20 and 300 N in a dry state and in distilled water prior to fracture testing. The cyclic loading had a force profile in the form of a sine wave at 2 Hz.<sup>16</sup> The specimens were then tested to failure as with the first subgroup. The data was statistically analyzed using oneway ANOVA. The factors were the two cements and the three testing modes. Means and standard deviations were calculated for each subgroup. Significant differences were evaluated using Tukey HSD test. All statistical testing was performed with  $\alpha =$ 0.05.

## Results

The mean values of the fracture loads of the crown specimens (nine subgroups) luted with composite resin cement are shown in Figure 2 and Table 1. The mean values of the fracture loads of the crown specimens (nine subgroups) luted with resin-modified glass ionomer cement are shown in Figure 3 and Table 2.

For the Variolink II resin cement groups, fracture loads of the In-Ceram Alumina, IPS Empress 2, and Cergogold specimens without fatigue were  $1528 \pm 238$  N,  $1412 \pm 153$  N, and  $947 \pm 144$  N, respectively. The fracture loads of In-Ceram and IPS Empress 2 were significantly greater than that of Cergogold ( $p \le 0.05$ ). No statistical difference was found between In-Ceram and IPS Empress 2 ( $p \le 0.05$ ).

The critical fracture loads for In-Ceram Alumina and IPS Empress 2 fatigued in a dry environment were  $1111 \pm 198$  N and  $1071 \pm 75$  N, respectively, which were statistically greater ( $p \le 0.05$ ) than that of Cergogold (698  $\pm 201$  N). The critical fracture loads of In-Ceram Alumina (843  $\pm 80$  N) and IPS



**Figure 2** Mean fracture loads of specimens luted with a resin cement and fractured under three conditions.

Empress 2 (895  $\pm$  56 N) specimens fatigued in a wet environment did not differ statistically from each other (p > 0.05), but they were statistically greater ( $p \le 0.05$ ) than Cergogold specimens (585  $\pm$  200 N).

For resin-modified glass ionomer cement, the critical loads of the In-Ceram Alumina, IPS Empress 2, and Cergogold were 1182  $\pm$  203 N, 1154  $\pm$  233 N, and 646  $\pm$  108 N, respectively. The critical fracture loads of In-Ceram and IPS Empress 2 specimens did not differ significantly (p > 0.05) from each other but were greater than that of Cergogold specimens statistically ( $p \leq 0.05$ ). After fatiguing in a dry environment, the critical fracture loads of In-Ceram Alumina (926  $\pm$  127 N) and IPS Empress 2 (868  $\pm$  67 N) specimens were not different from each other statistically ( $p \leq 0.05$ ); however, they were statistically greater ( $p \leq 0.05$ ) than Cergogold specimens (569  $\pm$  209 N). The same trend occurred in the wet environment, where the critical loads of In-Ceram Alumina (710  $\pm$  122 N) and IPS Empress 2 (760  $\pm$  70 N) specimens were statistically greater ( $p \leq 0.05$ ) than those of Cergogold specimens (512  $\pm$  176 N).

Within each ceramic type, the critical loads of specimens without fatigue were statistically superior ( $p \le 0.05$ ) to those fatigued in a dry environment. The fracture loads of specimens fatigued in a dry environment were statistically greater than those fatigued in distilled water ( $p \le 0.05$ ) (Figs 2 and 3). This

is true for both composite resin cement and resin-modified glass ionomer cement. Within the two cements studied, the critical fracture loads of the specimens using composite resin cement were statistically greater than those using resin-modified glass ionomer cement ( $p \le 0.05$ ) (Tables 1 and 2).

## Discussion

The null hypotheses in this study were: (1) critical loads of the crowns using the two cements are equal; (2) critical loads of the crowns tested in the three conditions, that is, no fatigue and fatigue in dry and wet environments, are equal; (3) there is no statistically significant difference in fracture loads for the three ceramic materials using the same cement and the same environmental condition. Our results indicate that all the null hypotheses were rejected. The critical loads of In-Ceram Alumina, IPS Empress 2, and Cergogold crowns cemented with a composite resin cement and resin-modified glass ionomer cement were affected by the type of cements and fatigue conditions. Even though the results of this study may not be directly related to the clinical situation, the current findings seem to correlate well with in vitro studies assessing the performance of ceramic restorations, as shown in the literature.<sup>16,20</sup> Earlier studies have shown that the thickness of the final restoration is

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Ceramic type	No fatigue		Fatigued and fracture tested in dry environment		Fatigued and fracture tested in distilled water	
	Mean	SD	Mean	SD	Mean	SD
In-Ceram	1528ª	238	1111ª	198	843ª	80
IPS Empress 2	1412 <sup>a</sup>	153	1071 <sup>a</sup>	75	895ª	56
Cergogold	947 <sup>b</sup>	144	698 <sup>b</sup>	201	585 <sup>b</sup>	200

Means followed by the same letters within each column indicate no statistical difference at 95% confidence level (p > 0.05). SD = standard deviation.



Figure 3 Mean fracture loads of specimens luted with a resin-modified glass ionomer cement and fatigued in dry or wet environments.

important. Small variations in thickness can affect the strength of the restoration.<sup>21,22</sup> In this study, the dimensions of the copings were carefully controlled by a device and a wax dipping unit for IPS Empress 2 and Cergogold and by a sculpting device, similar to that used in a previous study for In-Ceram Alumina.<sup>19</sup> Moreover, a split brass model was used for fabricating a stratification portion of the crown with feldspathic porcelain for each ceramic system; however, evaluating the fatigue behavior of dental ceramics with controlled specimen shapes and dimensions, that is, bar or disc specimens, can provide more definitive data. Ritter<sup>23</sup> derived the flexural strength equation for materials tested under constant stress rate as

$$\sigma_f^{n+1} = B\left(n+1\right) S_i^{n-2} \dot{\sigma} \tag{1}$$

where  $\dot{\sigma}$  is the stress rate, and  $S_i$  is the inert (moisture free) flexural strength. By performing a regression of  $\ln \sigma_f$  versus  $\ln \dot{\sigma}$ , a linear model of best fit can be constructed and used to estimate the subcritical crack growth parameters, *n* and *B*.

The current results showed that, for all three ceramic systems and three fatigue conditions, the critical loads of the crowns using resin cement exhibit greater values than those using resin-modified glass ionomer cement. The ability of the resin cement to provide a greater critical load for all-ceramic crowns than for conventional cements has been evaluated previously using stainless steel dies, and the fracture resistance

was improved when the crowns were cemented with resin cement.<sup>24</sup> Moreover, laboratory tests examining fracture loads of dentin-bonded crowns have been conducted using a standardized preparation similar to the one in the current study.<sup>16,25</sup> The results indicated that luting the crown with dual-cure resin cement produces significantly greater fracture load than conventional cements.<sup>26,27</sup> This may partially be explained by the lower bond strength between ceramics and glass ionomer resin cement.<sup>28</sup> In addition, the use of the resin-based luting material may increase the strength of ceramic by creating compressive stresses in the ceramic from polymerization shrinkage of the resin luting material and by crack bridging.<sup>29,30</sup> When all specimens were maintained in distilled water at least 24 hours before the tests, the resin-modified glass ionomer cement could expand on setting under moist environments. This could increase the propagation of cracks present in the ceramic structure.<sup>31</sup>

In this study, a significant decrease in critical load was observed when all ceramics were fatigued. Ceramic crown specimens fatigued in distilled water had the lowest fracture load values (Figs 2 and 3) within the testing environment groups. Specimens that had not been fatigued had the greatest fracture load values (Figs 2 and 3). The findings of this study are in agreement with what Sherrill and O'Brien<sup>32</sup> found for dental feldspathic porcelain. They tested porcelain in water and in dry conditions and found that only the wet condition decreased the

Table 2 Fracture	loads of specimens	luted with a r	resin-modified	glass ionomer	cement (N)
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Ceramic type	No fatigue		Fatigued and fracture tested in dry environment		Fatigued and fracture tested in distilled water	
	Mean	SD	Mean	SD	Mean	SD
In-Ceram	1182ª	203	926ª	127	710 <sup>a</sup>	122
IPS Empress 2	1154ª	233	868ª	67	760 <sup>a</sup>	70
Cergogold	646 <sup>b</sup>	108	569 <sup>b</sup>	209	512 <sup>b</sup>	176

Means followed by the same letters within each column indicate no statistical difference at 95% confidence level (p > 0.05). SD = standard deviation. final critical load. Furthermore, Sobrinho et al<sup>16</sup> demonstrated a decrease in strength after fatigue in a wet environment for In-Ceram and IPS Empress ceramic when compared with the dry condition. Mechanical fatigue alone could not degrade the interface between the ceramic materials and the cements as much as it could for the wet condition with the presence of stress corrosion. In addition, subcritical crack growth (SCG) significantly decreases survival time of dental ceramics.<sup>33</sup> SCG is crack propagation at stress intensity factor (K<sub>I</sub>) levels lower than the critical stress intensity factor, or fracture toughness (K<sub>IC</sub>).<sup>34</sup> Long-term or repetitive low-level loading may cause preexisting subcritical flaws to slowly grow until failure occurs at a level of loading insufficient to cause failure of the dental ceramic prosthesis before fatigue.

In this study, In-Ceram and IPS Empress 2 crowns had significantly greater fracture loads than Cergogold crowns tested in all conditions. The reason for the difference could be the composition of the materials. It has been shown that the increase in strength or toughness of a material is categorized as crack-tip deflection, crack-tip shielding, and crack bridging. In addition, the increase in strength can be achieved by increasing the proportion of dispersed ceramic crystals.<sup>35</sup> IPS Empress 2 is a lithium-disilicate-reinforced glass ceramic and has a crystallinity value of about 70  $\pm$  5% by volume.<sup>8</sup> The increased crystalline content forms a tighter, interlocking structure that significantly increases strength and fracture toughness. In-Ceram Alumina has a composition of approximately 82%  $Al_2O_3$  crystals, and its high strength has been explained by the high content of crystals associated with a glass that fills the spaces among them, which provides a homogeneous, bubblefree core material.<sup>16</sup> Cergogold has a composition mainly based on leucite, but the crystalline content has not been well quantified (the manufacturer claims that it has around 30% crystals). We suspect that Cergogold could be more similar to IPS Empress, which has a lower strength, than that of IPS Empress 2.

The limitation of this study could be the preparation procedure used, which does not reproduce clinical preparations in shape. In the clinic, it is difficult to control the thickness of ceramics. Despite the differences in preparation, the use of bovine teeth and a standardized preparation provided a reproducible testing base.

# Conclusion

Within the limitations of the study the following conclusion were made:

- (1) The ceramic crowns cemented with a composite resin cement resulted in fracture loads significantly greater  $(p \le 0.05)$  than those cemented with resin-modified glass ionomer cement.
- (2) Fracture loads of ceramic crowns decreased significantly after cyclic fatigue testing.
- (3) Fatigue testing in distilled water showed statistically lower  $(p \le 0.05)$  fracture loads of ceramic crown specimens than those tested in a dry environment.

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