

# Influence of different adhesive resin cements on the fracture strength of aluminum oxide ceramic posterior crowns

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**Statement of problem.** The influence of different types of adhesive resin cements on the long-term prognosis of aluminum oxide ceramic posterior crowns is unclear.

**Purpose.** The purpose of this study was to evaluate the fracture resistance of aluminum oxide ceramic on maxillary posterior crowns cemented with different resin luting agents before and after cyclic thermomechanical loading.

**Material and methods.** Forty-eight maxillary first molars were prepared and restored with standardized aluminum oxide ceramic (In-Ceram Alumina) crowns. The test specimens were randomly divided into 3 groups (n=16). The crowns were luted with an acrylic resin cement (Super-Bond C&B, control, Group SB) and 2 composite luting agents (Panavia F, Group PV; and Rely X Unicem, Group RX). Half of the specimens were exposed to thermomechanical fatigue in a masticatory simulator. All specimens were tested for fracture strength (N) using quasistatic loading. The Wilcoxon rank sum test was used to compare the fracture strength ( $\alpha=.05$ ).

**Results.** All specimens survived the exposure to the simulator. The following median fracture strength values were obtained without/with thermomechanical fatigue loading: Group SB, 2726 N/2673 N; Group PV, 2520 N/2083 N; and Group RX, 2036 N/2369 N. The fracture strength in Group PV after thermomechanical fatigue loading was significantly lower compared to the fracture strength in Group PV without artificial aging ( $P=.016$ ), as well as significantly lower compared to Group SB with artificial aging ( $P=.003$ ).

**Conclusion.** Within the limitations of this study, all tested cements are capable of successfully luting aluminum oxide ceramic crowns. The fracture strength of Group PV after artificial aging was comparatively low. (J Prosthet Dent 2004;92:359-64.)

## CLINICAL IMPLICATIONS

*The results of this in vitro study show that the selection of the adhesive resin cement may influence the fracture strength of aluminum oxide ceramic posterior crowns.*

The increase of patients' desire for esthetics has resulted in the use of all-ceramic restorations in the posterior region as well as the anterior region. The acceptance of all-ceramic restorations has increased because of their inherent esthetics, excellent biocompatibility, and improved physical properties.<sup>1-3</sup> Several types of all-ceramic materials have been developed for posterior

crowns, including conventional powder slurry ceramics,<sup>4</sup> castable ceramics,<sup>5,6</sup> leucite-reinforced ceramics,<sup>7,8</sup> aluminum oxide ceramics,<sup>9-11</sup> and zirconium oxide ceramics.<sup>12,13</sup> However, all-ceramic materials are brittle and weak when placed under tensile and torsional stress.<sup>14</sup> Furthermore, the restorations in the posterior region must tolerate high masticatory stresses. Physiologic maximal posterior masticatory forces may vary up to 880 N, depending on facial morphology and age.<sup>15,16</sup> In-Ceram Alumina (Vita Zahnfabrik, Bad Sackingen, Germany) is a glass-infiltrated aluminum oxide ceramic material that has been used for the fabrication of frameworks of crowns and fixed partial dentures in the anterior as well as the posterior regions.<sup>9-11,17,18</sup> It has been suggested that all-ceramic restorations should be bonded to tooth structure with adhesive resin cements to achieve adequate stability of the restorations<sup>3,19-21</sup> and fracture resistance of the restorations

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and the abutment tooth<sup>22-24</sup> and to prevent postoperative sensitivity.<sup>25-27</sup> This also applies to In-Ceram Alumina posterior crowns.<sup>11,28</sup> However, there is some evidence that adhesive cementation may not be necessary for the long-term clinical success of In-Ceram posterior crowns.<sup>10,29-31</sup> Clinical studies have not yet compared the influence of different luting agents on the clinical performance of such restorations.

With regard to adhesive cementation of aluminum oxide all-ceramic crowns, 2 different interfaces and an intermediate layer should be considered: (1) the tooth-cement interface, (2) the ceramic-cement junction, and (3) the cement layer itself. Most adhesive resin luting agents have adequate dentin and enamel bonding properties.<sup>32,33</sup> Several investigators<sup>34,35</sup> have also reported that the use of resin luting agents provides sufficient bond strength to glass-infiltrated aluminum oxide ceramic material. However, problems regarding the long-term bond durability at the tooth-resin and resin-ceramic interfaces have been reported.<sup>36-42</sup> Regarding the cement layer itself, adhesive resin cements have advantageous mechanical properties compared to conventional cements.<sup>43-46</sup>

Clinical durability of the restorations can only be evaluated through *in vivo* studies, but such studies are time consuming, expensive, and difficult to standardize. To simulate clinical conditions in the laboratory, masticatory simulators were developed to imitate physiologic thermomechanic fatigue loading.<sup>47-49</sup> The fracture resistance of all-ceramic crowns is dependent on thermomechanical fatigue in a masticatory simulator.<sup>50-52</sup> Use of a masticatory simulator decreased the fracture strength of all-ceramic crowns.<sup>51,52</sup>

The aim of this *in vitro* study was to evaluate the fracture strength of In-Ceram Alumina posterior crowns cemented with 3 different adhesive resin cements before and after cyclic thermomechanical loading. The null hypothesis was that the fracture strength was equal for all cements, with and without artificial loading.

## MATERIAL AND METHODS

Forty-eight caries-free human maxillary first molars were selected.<sup>51,53</sup> The molars were cleaned by scaling and stored in 0.1% thymol solution. To imitate physiologic tooth mobility, all roots of the teeth were covered with a gum resin (Anti-Rutsch-Lack; Wenko-Wenselaar, Hilden, Germany) to simulate the periodontal membrane.<sup>54,55</sup> The teeth were embedded in specimen holders perpendicular to the horizontal plane using an autopolymerizing acrylic resin (Technovit 4000; Heraeus Kulzer, Wehrheim, Germany). To simulate the biologic width, the resin was extended to 2 mm below the cemento-enamel junction (CEJ).

Each tooth was prepared for a complete-coverage all-ceramic crown using a silicone index. An occlusal reduc-

tion of 1.8 to 2.0 mm was prepared, followed by a circular 1.2-mm-wide accentuated chamfer preparation using a diamond bur (80- $\mu$ m grit). The final preparation resulted in a taper of 4-6 degrees and an approximate abutment height of 4.5 mm. All sharp line angles were rounded, and all margins were finished 0.5 mm apical to the CEJ using fine diamond burs (30- to 40- $\mu$ m grit). All preparations were done by hand, no milling device was used. Impressions of the prepared abutments were made using a putty-wash technique. A vinyl polysiloxane impression material (Monopren; Kettenbach, Eschenburg, Germany) was syringed around the abutments, and putty material (Twinduo; Picodent, Wipperfurth, Germany) was used in the custom-made impression tray. Dies were fabricated using dental stone (Fujirock II; GC Europe, Leuven, Belgium).

The ceramic frameworks were fabricated using glass-infiltrated aluminum oxide all-ceramic blanks (In-Ceram Alumina; Vita Zahnfabrik) using CAD/CAM technology (Wol-Ceram; Wol-Dent, Ludwigshafen, Germany). Each core had a thickness of 0.5 mm, except at the occlusal surface, where the core material was 0.7 mm thick. After verification of the framework dimensions with a caliper (SU metal caliper; Schuler-Dental, Ulm, Germany), glass infiltration of the ceramic material was performed (Vacumat 40; Vita Zahnfabrik) at 1100°C. Excess glass was removed using a coarse-grit diamond bur followed by airborne-particle abrasion with aluminum oxide (Al<sub>2</sub>O<sub>3</sub>) (50  $\mu$ m), using 4.0 bar compressed air. All copings were then veneered and glazed with feldspathic porcelain (Vita VM7; Vita Zahnfabrik). The intaglio surfaces of the crowns were airborne-particle abraded with 50- $\mu$ m Al<sub>2</sub>O<sub>3</sub> particles at an air pressure of 2.5 bar and ultrasonically cleaned in 96% isopropyl alcohol.

The 48 crowns and the corresponding casts were divided at random into 3 groups (n=16) and assigned to 1 of the following cement groups: Super-Bond C&B (Group SB), Panavia F (Group PV), and Rely X Unicem (Group RX) (Table I). All crowns were cemented on the abutments according to the manufacturer's recommendations. During cementation, the crowns were secured in place with finger pressure for 5 minutes. After crown placement, all specimens were stored in a 0.1% thymol solution for 24 hours before testing.

Half of the specimens (n=8) in each group were subjected to a quasistatic loading at a cross-head speed of 1.5 mm/min in a servohydraulic universal testing machine (Z010/TN2S; Zwick, Ulm, Germany) until fracture. A perpendicular load was applied to the occlusal surface of the crown using a tin foil to distribute the force. The applied force (N) was recorded on an x-y recorder, with failure defined as a deviation from graphic linearity. The remaining 8 specimens from each group were exposed to 1,200,000 cycles of thermomechanical fatigue in a

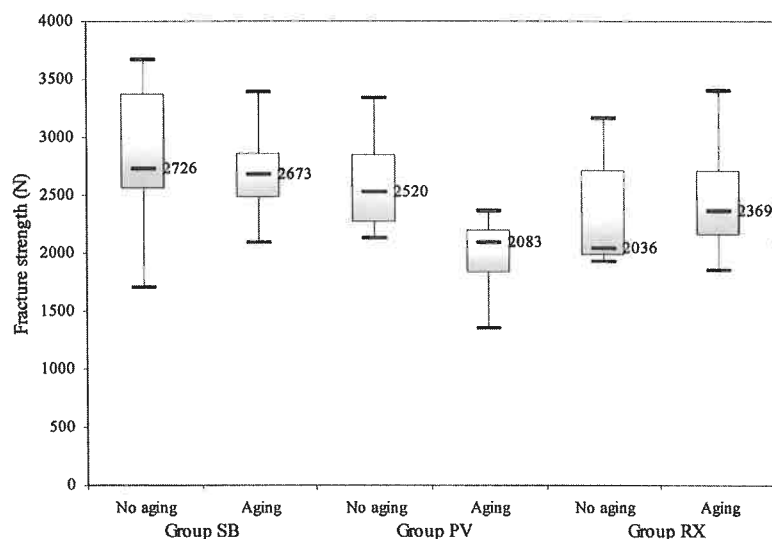


Fig. 1. Box plots of fracture strength values of 6 groups. Line in each box represents median value.

Table I. Adhesive resin cements

Group	Cement	Main component	Ceramic primer	Manufacturer
SB	Super-Bond C&B	PMMA, MMA, 4-META, TBB	Porcelain liner M	Sun Medical, Shiga, Japan
PV	Panavia F	Filler (78%), MDP, dimethacrylates, initiator	Clearfil SE Bond, porcelain bond activator	Kuraray Medical, Tokyo, Japan
RX	Rely X Unicem	Filler (72%), dimethacrylates, methacrylated phosphoric ester	Rely X Ceramic Primer	3M ESPE, Seefeld; Germany

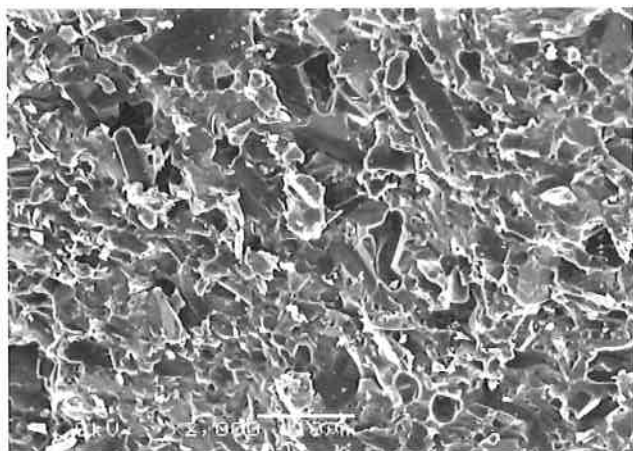
PMMA, Polymethyl-methacrylate; MMA, methyl-methacrylate; 4-META, 4-methacryloyloxyethyl trimellitate anhydride; TBB, tri-*n*-butyl borane; MDP, 10-methacryloyloxydecyl di-hydrogen phosphate.

computer-controlled dual axis masticatory simulator (Willytec; München, Germany).<sup>47</sup> The force was applied occlusally in the central fossa of the crown at a frequency of 1.3 Hz<sup>56</sup> using a ceramic ball with a diameter of 6 mm (Steatite; Hoechst Ceramtec, Wunsiedel, Germany). A dynamic load of 49 N was chosen to simulate a load within the physiologic range.<sup>57</sup> During testing, all specimens were subjected to simultaneous thermal cycling between 5°C and 55°C for 60 seconds each, with an intermediate pause of 12 seconds, maintained by a thermostatically controlled liquid circulator (Haake, Karlsruhe, Germany).<sup>47,55</sup> All specimens that did not fracture during dynamic loading and thermal cycling were loaded compressively in the universal testing machine (Z010/TN2S; Zwick) until fracture. After fracture testing, the intaglio surface of the fractured crown and the dentin of the abutment were ion coated with platinum (ESC-101; Elionix, Tokyo, Japan). The surfaces of the ion-coated specimens were observed under a scanning electron microscope (JSM-5610LV; Jeol, Tokyo, Japan). The results of fracture strength were displayed by using box plots. The Wilcoxon rank sum test was used to compare fracture strength between groups, and without and after exposure to the artificial mouth ( $\alpha=.05$ ).

## RESULTS

All specimens survived 1,200,000 cycles of dynamic loading and thermal cycling in the artificial oral environment. The following median fracture strength values were obtained without/with thermomechanical fatigue loading: Group SB, 2726 N/2673 N; Group PV, 2520 N/2083 N; and Group RX, 2036 N/2369 N (Fig. 1). The intragroup comparison of fracture strength in Group PV after the thermomechanical fatigue and without artificial aging was significant ( $P=.016$ ). For Groups SB and RX the intragroup comparison was not significant. All between-group comparisons without artificial aging were not significant. After artificial aging the fracture strength in Group SB was significantly higher than in Group PV ( $P=.003$ ).

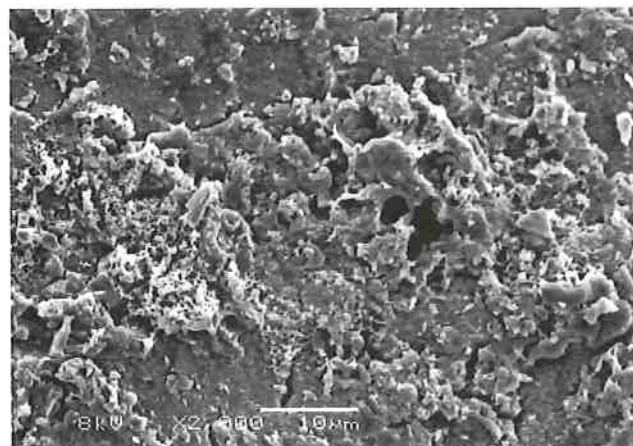
When examining the fracture patterns of the crowns, none of the fractures occurred at the ceramic-resin interface or within the ceramic material. Cohesive failure within only the resin cement was observed throughout the surface in all of Group SB specimens after exposure to fatigue loading (Fig. 2). The fractured surface in the resin cement was rough. For Group PV with fatigue loading, adhesive failures between the resin cement and dentin primed with the self-etching primer were



**Fig. 2.** SEM observation of fractured surface of Group SB with fatigue loading (original magnification  $\times 2000$ ).



**Fig. 3.** SEM observation of fractured surface of Group PV with fatigue loading (original magnification  $\times 2000$ ).



**Fig. 4.** SEM observation of fractured surface of Group RX with fatigue loading (original magnification  $\times 2000$ ).

observed. No exposed dentinal tubules could be observed, and smear layer-like agglomerates were attached to the dentin surface (Fig. 3). The fractured surface was mostly smooth. Failure of Group RX with fatigue loading occurred between the hybrid layer and the resin cement, and a small amount of remaining resin cement was observed (Fig. 4). The surface to which remnants of the resin cements were attached appeared relatively smooth.

## DISCUSSION

The stated null hypothesis in this study, that fracture strength is equal for all cements, with and without artificial loading, was rejected. The fracture strength of In-Ceram Alumina crowns cemented with an adhesive resin cement were affected by the different types of cements and the cyclic thermomechanical loading. Although the findings from this *in vitro* study are not directly transferable to the clinical situation, the process has been shown to correlate well with clinical studies assessing the performance of restorations over 5 years.<sup>48,49</sup>

Panavia F and Rely X Unicem consist of multifunctional phosphoric acid dimethacrylate-modified monomers, such as Bis-GMA, and inorganic fillers of fine glass and silica.<sup>45</sup> These resin cements exhibit high compressive strength and diametral tensile strength.<sup>43</sup> However, no inorganic filler is contained in Super-Bond C&B, as the primary ingredient is referred to as 4-META/MMA-TBB resin.<sup>33,45</sup> The compressive and diametral tensile strengths of this cement are impossible to measure because of its low modulus of elasticity.<sup>33</sup> In the present study, although there was no significant difference in fracture strength without fatigue loading among the 3 test groups, Group SB demonstrated the highest values. For a restoration to function satisfactorily over many years, it has been postulated that the luting agent must have sufficient physical strength to resist fracture and long-term cyclical fatigue stresses.<sup>43</sup> However, the findings from the present study conflict with other data.<sup>43</sup> It may be assumed that the ductile resin cement thus functions as a shock absorber so that it can distribute forces during the fracture testing on the tooth-cement-ceramic complex. In addition, Super-Bond C&B contains long flexible chains of high molecular weight, which tend to result in higher fracture toughness values when compared with highly cross-linked brittle materials contained in other composite cements.<sup>45,46</sup> Plastic deformation delays the onset of brittle fracture, resulting in higher fracture toughness values.<sup>45,46</sup>

In the current study, the survival rate of the In-Ceram Alumina crowns after exposure to the simulated oral environment was 100%. Moreover, the median fracture strength values before and after exposure to the simulator reached levels higher than the physiological maximum occlusal force.<sup>58</sup> Thus, all tested crowns possess the potential to withstand physiologic occlusal forces. The statistical analysis demonstrated a significant influence of cyclic fatigue loading on the fracture strength

of crowns cemented with Panavia F. Fatigue loading reduced the fracture strength by approximately 25% for crowns cemented with Panavia F. Fatigue loading in the simulator did not significantly affect the fracture strength of crowns luted with Super-Bond C&B and Rely X Unicem. Ideal situations in the tooth-cement and cement-ceramic interfaces and the cement layer are not always obtained in clinical practice, due to such factors as the occlusal force, thermal stress, and saliva immersion. These *in vivo* factors may negatively affect the dentin-cement and cement-ceramic interfaces and the cement layer,<sup>41</sup> which may explain the decreased durability of crowns cemented with Panavia F.

In the current study, the fracture strength of In-Ceram posterior crowns with fatigue loading was significantly different among the 3 cement groups. Leevailoj et al<sup>31</sup> reported that the type of luting agent had no significant effect on the compressive fracture loads of In-Ceram premolar crowns. These conflicting results mainly arise from different experimental methods; no cyclic fatigue loading was performed in the earlier study.<sup>31</sup> Thus, different types of adhesive resin cements may affect fracture strength of In-Ceram crowns in long-term clinical observation.

The SEM observations in the current study indicated that the tooth-cement interface appeared to be the weaker link rather than the ceramic-cement interface, where no fractures occurred. The fracture of Group PV with fatigue loading occurred through adhesive failure between the resin cement and the primed dentin surface, and the fractured surface was mostly smooth. These findings indicate that during the process of crack propagation the amount of breaking elongation at the crack tip was small.<sup>46</sup> Therefore, these results suggested that the resistance of the bonding of Group PV is low. On the other hand, SEM photographs of Group SB with fatigue loading showed cohesive failure only within the resin cement; none of the crown fractures occurred between the resin cement and the dentin interface or within the dentin. In addition, the fractured surfaces were rough. These results illustrate that the amount of the breaking elongation at the crack tip of this cement increased.<sup>46</sup> Although there were no significant differences in fracture strength, the SEM analysis showed Group RX with fatigue loading had a different type of fracture pattern. Failure of Group RX occurred between the hybrid layer and the resin cement, and a small amount of remaining resin cement was observed. The surface to which remnants of the resin cements were attached appeared relatively smooth. The hybrid layer, which is the weakest link to achieve durable long-term bonding in the cement-dentin complex,<sup>41</sup> was not affected in Group RX specimens. The bonding conditions of Group RX appeared relatively stable.

The design of this *in vitro* study has several limitations, making it difficult to compare the results

with the clinical situation. Natural teeth were used as abutments in this study. The primary disadvantage of natural teeth is the relatively large variation in size and mechanical parameters,<sup>53</sup> often resulting in large standard deviations. Several authors used steel or resin dies for the fracture testing of crowns.<sup>21,24</sup> The advantages of using dies are the possibility of a standardized preparation and the identical physical properties of materials. However, using natural teeth for the fracture testing of crowns has been recommended,<sup>53</sup> as abutments made of steel or resin do not reproduce the actual force distribution that occurs on crowns cemented to natural teeth. In the artificial oral environment used in this study, the load was primarily applied axially with regularity and identical force. However, the direction and the power of masticatory forces are variable clinically. In the current study, only the fracture strength values and SEM observation were investigated. However, in order to comprehensively examine the prognosis of In-Ceram crowns, other evaluation methods such as marginal adaptation or dye-penetration tests should be used. Furthermore, clinical trials are necessary to validate the results of the present *in vitro* study.

## CONCLUSIONS

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

1. All cements tested were capable of successfully luting aluminum oxide ceramic crowns.
2. The fracture strength of crowns luted with Panavia after artificial aging was significantly lower than Panavia specimens that were not artificially aged.

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