

# Fracture resistance of CAD/CAM-generated composite resinbased molar crowns

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# Fracture resistance of CAD/CAM-generated composite resin-based molar crowns

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#### Abstract

The aim of this study was to investigate whether different fabrication processes i.e., a CAD/CAM system or the manual build-up technique, affect the fracture resistance of composite resin-based crowns. Lava Ultimate (LU), Estenia C&B (EC&B), and lithium disilicate glass-ceramic IPS e.max press (EMP) were used. Four types of molar crowns were fabricated: 1) CAD/CAM-generated composite resin-based crowns (LU crowns), 2) manually built-up monolayer composite resin-based crowns (EC&B-monolayer crowns), 3) manually built-up layered composite resin-based crowns (EC&B-layered crowns), and 4) EMP crowns. Each type of crown was cemented to dies and the fracture resistance was tested. EC&B-layered crowns showed significantly lower fracture resistance than LU and EMP crowns though there was no significant difference in flexural strength or fracture toughness between LU and EC&B materials. The micro-CT and fractographic analysis showed that decreased strength likely resulted from internal voids in the EC&B-layered crowns introduced by the layering process. There was no significant difference in fracture resistance among LU, EC&B-monolayer and EMP crowns. Both types of composite resin-based crowns showed fracture loads >2000 N, which is higher than the molar bite force. CAD/CAM-generated crowns without internal defects, may be applied to molar regions with sufficient fracture resistance.

Key words: composite resin; CAD/CAM; lithium disilicate; micro-CT; fracture resistance

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#### Introduction

Metal-ceramic, all-ceramic and metal-cast crowns are successfully used in molar region (1, 2). All-ceramic crowns possess sufficient esthetics and adequate strength to function in molar region (3-5) though the treatment costs may be relatively high. As for metal-ceramic crowns, the esthetics may be challenging because metal framework will reduce light transmission through the crown, and has a risk of discolored margins. In addition, concerns have been raised about adverse effects caused by metal alloys (6, 7). In this context, application of composite resin-based crowns, here after referred to as composite crowns, in molar region has been proposed as an inexpensive alternative with proper esthetics (8-11).

Several studies on the clinical performance of composite crowns have been conducted (12-14). Rammelsberg et al. (13) found that composite crowns (68 posterior and 46 anterior crowns) made by manual build-up technique (Artglass, Heraeus Kulzer, Hanau, Germany) exhibited acceptable survival rate of 96% after 3 years. Contradictory, Vannorbeek et al. (14) reported a 3-year survival rate of 87.9% for composite crowns (40 posterior and 19 anterior crowns), which were CAD/CAM-generated composite copings veneered with another composite material (GN-1 and Gradia, GC Tokyo, Japan), and even survived crowns suffered from complications. The main reason for failures in both studies was fracture, suggesting that the strength of composite resin crowns is one of the most important properties to obtain a good clinical result.

The mechanical properties of composite resin-based materials have been improved over the past decades for example by increasing the amount of inorganic fillers incorporated in the resin matrix (15). Up to 92 wt.% ( $\approx$  70-75 vol.%) have been achieved with increased

mechanical strength according to the manufacturer's data for Estenia C&B (Kuraray Noritake Dental, Tokyo, Japan). In addition to the supposed improvement of material, the fabrication process of restorations has been developed using CAD/CAM technique. Composite restorations often contain internal defects (*i.e.* voids), especially when spatulated, resulting in decreased strength (16-19). To minimize this risk, CAD/CAM technique has been applied to make indirect composite restorations. In that system, industrially manufactured composite resin blocks with a high degree of homogeneity are used (20, 21). However, it is still unclear if that advantage will improve the fracture resistance of composite crowns.

Previous *in vitro* studies (11, 22) have shown that composite crowns could have higher fracture resistance than the average maximum bite force (700-900 N) reported in the literature (23-26). Ohlmann et al. (27) demonstrated that the fracture resistance of composite crowns (Artglass) depended on the occlusal thickness, type of cements and convergence angle of abutments. Crowns with an occlusal thickness of 1.3 mm luted with a composite resin-based cement to abutment teeth prepared with a convergence angle of 4°, showed a mean fracture load of approximately 2200 N. It seems therefore reasonable to assume that composite crowns can withstand the forces in the molar region. In addition, evaluation of fracture load of composite crowns comparing with that of all-ceramic crowns successfully used in the clinical situation under the same test condition will probably give additional information. To the knowledge of the authors no studies have been conducted yet in that matter.

Based on the background, it was hypothesized that CAD/CAM-generated composite crowns should possess fewer voids than manually built-up ones, and as such, should show

higher fracture resistance, which might be comparable to that of lithium disilicate crowns. The purpose of the present study was 1) to analyze the influence of internal voids related to the fabrication technique on the fracture resistance of composite crowns, and 2) to compare the fracture resistance of CAD/CAM-generated and manually built-up composite crowns with that of monolithic lithium disilicate crowns.

#### Materials and methods

## Flexural strength test

Materials tested in this study and their abbreviations are shown in Table 1. Ten bar-shaped specimens (22.3×2×2 mm) were fabricated from each material. Lava Ultimate (composite resin blocks for the CAD/CAM technique), Estenia C&B (composite resins pastes for manual build-up technique) and IPS e.max Press (lithium disilicate glass-ceramic for the press technique) were tested for comparison. LU specimens were cut from blocks using a cutting device (IsoMet 400, Buehler, Germany). EC&B-OD, EC&B-D and EC&B-E specimens were formed using a split stainless steel-mold and glass plates. Then, the specimens were cured by photopolymerization for 5 min using a laboratory light curing unit (α-Light II, Morita, Tokyo, Japan) equipped with a halogen lamp (360 W) and two fluorescent lights (27 W). After light curing, heat polymerization at 110°C for 15 min using a heat-curing unit (KL-400, SK medical electronics, Tokyo, Japan) was performed according to the manufacturer's instruction. For preparation of EMP specimens, bar-shaped wax patterns were made using the stainless steel-mold. The wax patterns were invested (Press Vest Speed, Ivoclar/Vivadent) and the investment was pre-heated at 850°C for 45 min followed by pressing in a press furnace

(Programat EP 5000, Ivoclar/Vivadent). After divesting, the specimens were ultrasonically cleaned in IPS e.max Press Invex Liquid (Ivoclar/Vivadent). All specimens were polished with silicon carbide papers up to #1000.

The specimens were inspected using micro-computed tomography (micro-CT, ScanXmate-D225RSS270, Comscantecno, Kanagawa, Japan) before the flexural strength test. The measurement conditions were: voltage; 100 kV (composite resin) vs. 90 kV (lithium disilicate), current; 220  $\mu$ A, resolution (voxel size) 31.8  $\mu$ m. Specimens containing voids with a diameter of >100  $\mu$ m (3 pixels in tomograms) in the middle area was excluded from the flexural strength test.

The flexural strength test was performed in a three-point bending test according to ISO 10477: 2004, "Dentistry-Polymer-based crown and bridge material" (28). The specimens were loaded at a crosshead speed of 0.5 mm/min and with a 15-mm support span in a universal testing machine (AG-IS, Shimadzu, Kyoto, Japan). Flexural strength and modulus of elasticity were calculated using the following equations:

$$\sigma=3FL/2bh^2$$

where  $\sigma$  is flexural strength (MPa), F is maximum load (N), L is length of support span (mm), b is specimen width (mm), and h is specimen thickness (mm).

$$E=[F_1/d]\times[L^3/4bh^3]$$

where E is modulus of elasticity (MPa),  $F_l/d$  (N/mm) is the slope of the linear portion of load-deflection line, L is the length of support span (mm), b is specimen width (mm), and h is specimen thickness (mm).

#### Fracture toughness test

Five bar-shaped specimens (22.3×3×4 mm) were fabricated from each material as described above. The 3 mm width face of each specimen received a starter notch with a depth of 1 mm in the middle vertically to the long axis using an automatic dicing saw (DFD64341, Disco, Tokyo, Japan). The notch was manually deepened using a razor with 6 μm diamond suspension to form V-notch. The depth of the V-notch was adjusted to be 1.4±0.1 mm using a stereomicroscope.

Fracture toughness test was performed in a three-point bending test according to ISO6872: 2008 "Dentistry-Ceramic materials. Annex A: Fracture toughness" (29). The specimen placed on two supports laying the face with the V-notch down was loaded at a crosshead speed of 0.5 mm/min and with a 16-mm support span in a universal testing machine (AG-IS). Fracture toughness was calculated using the following equations given in ASTM C1421-10 "Standard test methods for determination of fracture toughness of advanced ceramics at ambient temperature" (30).

$$K_{IC} = g[PmaxS_010^{-6}/BW^{3/2}][3(a/W)^{1/2}/2(1-a/W)^{3/2}]$$

$$g=[1.99-(a/W)(1-a/W)\{2.15-3.93(a/W)+2.7(a/W)^2\}]/[1+2(a/W)]$$

where  $K_{IC}$  is fracture toughness (MPa·m<sup>1/2</sup>), g is function of the ratio a/W, Pmax is maximum load (N),  $S_0$  is length of support span (m), B is side to side dimension of the test specimen perpendicular to the notch depth (m), W is top to bottom dimension of the test specimen parallel to the notch depth (m), and a is depth of V-notch (m).

#### Fabrication of dies and crowns

Tooth preparation and fabrication of dies were performed according to the protocol from previous studies (31). In brief, a plastic tooth model of mandibular right first molar (A5A-500, NISSIN, Kyoto, Japan) were prepared with a 1.0-mm chamfer width, minimal occlusal reduction of 1.6 mm and a convergence angle of 10° (Fig. 1). Then, 24 replicas of the abutment tooth (*i.e.* dies) were milled from composite resin blocks (Lava Ultimate).

Six crowns were fabricated for each group. For fabrication of the CAD/CAM-generated Lava Ultimate crowns (LU crown), the dies were scanned, and crowns were designed by double scan technique with additional scanning of the non-prepared tooth model. The cement space was set to be 70 μm. Thus, the minimum thickness of crown at occlusal surface was expected to be > 1.5 mm as a result of subtraction of cement space from occlusal reduction. Based on the design, six crowns were milled from composite resin-based blocks (Lava Ultimate). After milling, polishing was performed using a wheel brush together with polishing agent (Zircon-Brite, Dental Ventures of America, Corona, CA, USA).

Two types of manually built-up composite crowns were fabricated using Estenia C&B. The one consisted of layers of EC&B-OD, EC&B-D and EC&B-E (EC&B-layered crown) as recommended by the manufacturer. The other had only one layer of EC&B-D (EC&B-monolayer crown) (Fig. 1). A spacer was put on a die to obtain a cement space of about 70 μm. For fabrication of EC&B-layered crowns, Opacious dentin (EC&B-OD) was manually built-up to be about 0.3 mm in thickness. Preliminary photopolymerization was performed for 90 s in the light curing unit (α-Light II). To obtain identical shapes of Dentin (EC&B-D) and Enamel (EC&B-E) for all crowns, molds were prepared using a transparent silicone material (Memosil2, Heraeus Kulzer). The dentin mold was made using a wax pattern

that reproduced dentin shape while the enamel mold was made using a non-prepared tooth model. The wax pattern of dentin was evaluated and adjusted using the digital scanner so that the EC&B-E layer would become 0.5 mm in the occlusal surface. Using the mold, EC&B-D was built up on EC&B-OD. Preliminary photopolymerization was performed for 10 s with the mold and another 10 s without the mold. Then, EC&B-E was built up on EC&B-D in the same manner. For each layering procedure, modeling liquid was applied to bond each layer. Finally, the crowns applied with Air Barrier Paste (Kuraray Noritake Dental) was photopolymerized for 300 s followed by heat polymerization at 110 °C for 15 min in the heat-curing unit (KL-400). EC&B-monolayer crowns were also fabricated by the same manner as described but without layering. Only EC&B-D was built up on the die using the mold used for the layering of EC&B-E. In order to reduce technical errors, one trained operator performed the manufacturing.

For fabrication of monolithic lithium disilicate crowns (EMP crowns), wax patterns were prepared using a mold of a non-prepared tooth model. Investing, pre-heating, pressing and divesting were performed by the same protocol for preparation of bar-shaped specimens. After ultrasonic cleaning, the crowns were glazed (IPS e.max Ceram Glaze, Ivoclar/Vivadent) in the press furnace.

The internal voids in the crowns were non-destructively inspected using micro-CT device (ScanXmate-D225RSS270) before load-to-failure test. The measurement conditions for micro-CT were as follows: the voltage, 100 kV; the current,  $500 \mu A$ ; the resolution (voxel size),  $14.9 \mu m$ ; the rotation,  $360^{\circ}$ ; number of projections, 1200. The CT data was reconstructed using a software (coneCTexpress, Comscantecno), and then the reconstructed

images were analyzed using an image processing program, ImageJ, provided by the Research Services Branch of the NIH. The number of internal defects (*i.e.* voids) in the crowns was counted and the volume of each void was calculated according to the following formula;

$$V = \sum [(VA \times W) \times 10^{-9}]$$

where  $V \text{ (mm}^3)$  is total volume of void, VA is area of voids per tomogram ( $\mu\text{m}^2$ ), and W is slice width of tomography (14.9  $\mu\text{m}$ ).

#### Load-to-failure test of crowns

The crowns were cemented to the dies using a resin-based cement (Panavia F2.0, Kuraray Noritake Dental). The cementation was performed according to the manufacturer's instruction. During cementation, the crown seated on the die was placed under a static load of 20 N for 5 min in the universal testing machine (AI-GS) according to a previous report (32). In addition, air-barrier gel, Oxyguard (Kuraray Noritake Dental), was applied around the margin of the crown. After cementation, the crown-die samples were stored in distilled water 37°C for 24 h before the load-to-failure test.

The load-to-failure test was performed in the universal testing machine (AI-GS) according to the previous study (31). Briefly, a semi-spherical indenter (type 304 stainless steel) with 10 mm in diameter was placed in the central fossa of occlusal surface. A urethane rubber sheet (Thickness=2 mm, Shore A Hardness=90) was interspersed between the indenter and the occlusal surface to avoid contact damage (33). A preload of 20 N was applied vertically to the crown followed by compressive test at a crosshead-speed of 0.5 mm/min until fracture occurred. After the load-to-failure test, fractographic analysis was performed on two

randomly selected samples from each group. The samples were coated with a 15-nm gold layer and imaged with scanning electron microscope (SEM; JXA-8500F, JEOL, Tokyo, Japan).

#### **Statistics**

Statistical analyses were performed using JMP Pro 11.0.0 software (SAS Institute, Cary NC, USA). Significant differences (p<0.05) in the flexural strength, the modulus of elasticity, the fracture toughness and the fracture load for each type of crowns were analyzed by analysis of variance followed by Tukey-Kramer HSD multiple comparison test. Significant differences (p<0.05) in the number of voids and the volume of voids for each type of crowns were analyzed by Steel-Dwass test.

#### Results

#### **Mechanical properties of materials tested**

Micro-CT analysis showed that no void existed in the middle area of the bar-shaped composite specimens (LU, EC&B-OD, EC&B-D and EC&B-E) while the bar-shaped EMP specimens contained small voids. The voids in EMP were, however, smaller than φ100 μm. Thus, all specimens were subjected to the flexural strength test. The results of the flexural strength test are shown in Fig. 2a. EMP showed significantly higher flexural strength than LU and EC&B; whereas there was no significant difference between the latter two. As for the modulus of elasticity, EMP showed significantly higher value than LU and EC&B. In addition, EC&B had significantly higher modulus of elasticity than LU (Fig. 2b). Fracture toughness

for each material is displayed in Fig. 2c showing the same trend with the flexural strength.

## Micro-CT analysis of crowns

The micro-CT analysis revealed that no void existed within LU crowns. By contrast, EC&B-layered crowns, EC&B-monolayer crowns and EMP crowns contained voids (Fig. 3). The average number and total volume of voids per crown are summarized in Table 2. EC&B-monolayer crowns showed significantly lower number and volume of voids than EC&B-layered crowns and EMP crowns. Although EMP crowns contained voids more than EC&B-layered crowns, the total volume in EMP crowns was significantly lower than EC&B-layered crowns. The voids in EC&B-layered crowns were mainly localized in the interface between different layers; while no remarkable localization of voids in EMP crowns was observed. Since the volume of the crown was calculated to be 330 mm³ based on the micro-CT analysis, the fractions of voids for EC&B-layered, EC&B-monolayer and EMP crowns were 0.0062, <0.0001 and 0.0003%, respectively.

#### Load-to-failure test

The mean values (±SD) of fracture loads for LU crowns, EC&B-layered crowns, EC&B-monolayer crowns and EMP crowns were 2880 (154), 2182 (446), 2602 (290) and 2719 (250) N, respectively (Fig. 4). LU crowns and EMP crowns showed significantly higher fracture resistance than EC&B-layered crowns. There was no significant difference in the fracture load among LU crowns, EC&B-monolayer crowns and EMP crowns.

The fracture pattern along with the central groove on the occlusal surface and from

one approximal surface to the other was observed for each crown. In addition, the crowns fractured together with the dies. The SEM analysis showed no signs of Hertzian cone cracks in the occlusal surface (Fig. 5). Voids were observed in the fracture surface of EC&B-layered and EMP crowns. In all cases, primary fracture origin was located at occlusal surface (Fig. 5).

#### Discussion

The present study investigated the influence of fabrication techniques on the fracture resistance of composite resin-based crowns. The results showed that CAD/CAM-generated crowns (LU crowns) showed significantly higher fracture resistance than manually layered composite crowns (EC&B-layered crowns), although there was no significant difference in the flexural strength and the fracture toughness between the bar-shaped composite resins specimens used for each technique. The micro-CT as well as the fractographic analysis showed that decreased fracture resistance of manually built-up composite crowns likely resulted from internal voids introduced by the layering process. In addition, CAD/CAM-generated composite crowns (LU crowns) showed almost equivalent fracture resistance to monolithic lithium disilicate crowns (EMP crowns) despite that the flexural strength and the fracture toughness of the bar-shaped composite specimens were significantly lower than those of lithium disilicate.

In the present study, the preparation of materials and load-to-failure test were performed basically according to the recommendation for a clinically relevant preclinical test (34). The testing method has also been used in a previous study (31). It has been recommended that the elastic properties of die materials (abutment tooth) should be similar to

those of dentin to obtain clinically relevant fracture of crowns (35-37). Thus, Lava Ultimate, which has similar modulus of elasticity and Poisson's ratio to those reported for dentin, was used as a die material (31).

To compare the influence of fabrication technique on the fracture resistance of composite crowns, the materials used for each technique should have equivalent mechanical properties, such as flexural strength and fracture toughness, because they characterize the responses of material to loading and crack propagation, respectively (38, 39). Thus, the flexural strength and fracture toughness were firstly examined, and it was confirmed that there was no significant difference between the composite materials used (i.e. Lava Ultimate and Estenia C&B). Still, there are other factors that affects the fracture resistance of crowns in load-to-failure test, such as type of preparation, cement and crown thickness (27, 36, 40). To eliminate their effects, standardized dies and the same resin-based cement were used for all groups. For fabrication of crowns with the same thickness for each group, the identical shape of crowns was obtained by scanning the non-prepared tooth model in the CAD/CAM technique and by preparing a mold of the non-prepared tooth model for manually built-up technique. Taking these considerations into account, the difference in the fracture resistance between LU crowns and EC&B-layered crowns could be attributable to the fabrication techniques. Based on the micro-CT analysis, the EC&B-layered crowns contained voids inside the crowns along with the layers while no void was observed in LU crowns. In addition, there was no significant difference in the fracture resistance between LU crowns and EC&B-monolayer crowns, which contained only few voids. These findings suggest that internal voids within composite crowns may decrease the fracture resistance. Thus, it would

be beneficial to use CAD/CAM technique for fabrication of composite crowns to reduce the risk of fracture related to internal voids.

The manual build-up technique used in this study was in some degree different from the general technique. Composite crowns are rarely fabricated using molds though they were used to reproduce the identical outer shape of crowns for experimental purposes in this study. Thus, the operator was trained to get used to the fabrication process using the molds. Although the effort to reduce the technical errors was made, voids were still generated in the manually built-up crowns. This can also happen using the general technique without a mold because the layers are built up by instrumentation that will increase the risk of infusion of voids (17, 19). Based on the results of the present study it might therefore be important to consider the use of monolithic composite crowns in the molar regions where fracture resistance is probably more important than esthetics.

The composite crowns without layering (LU crowns and EC&B-monolayer crowns) showed high fracture resistance comparable to the lithium disilicate crowns tested (EMP crowns). Still, flexural strength and fracture toughness of the bar-shaped EMP specimens showed approximately two times higher values than those of the bar-shaped composite specimens. This may be explained by the relationship of the modulus of elasticity between the crown and die materials. In the case of the composite crowns, the crowns had the well matched modulus of elasticity with the die (*i.e.* LU). When composite crowns are bonded to dies possessing the similar modulus of elasticity, the crown-die complex functions as one integrated body showing higher fracture resistance than the crowns bonded onto metal dies (41). However, the EMP crowns had much higher modulus of elasticity than the die. It has

been demonstrated that the more elastic the die is, the lower the fracture resistance of all-ceramic crowns becomes (36, 42). Thus, the fracture resistance of LU crowns and EC&B-monolayer crowns would be comparable to that of EMP crowns. This may be also the case in the clinical situation since the die material used have comparable values of modulus of elasticity and Poisson's ratio as vital dentin (26).

The fractographic analysis showed no signs of Hertzian cone cracks, suggesting that contact damage of the steel indenter was successfully avoided (34). This was also supported by the fracture pattern observed along with the central groove but not at the loading points. The loading points are possible sites where the contact damage would occur. Regarding the defects on the fracture surface, several voids were observed in EC&B-layered crowns and EMP crowns. Thus, it is speculated that voids may result in lower fracture resistance of the crowns tested, especially EC&B-layered crowns. Additionally, it was observed that the primary fracture origin was located at the occlusal surface regardless of the type of the crowns. By contrast, it has been reported that clinically failed all-ceramic crowns fractured from the cervical area (43-46). Øilo et al. (33) demonstrated that the clinically relevant fracture mode could be reproduced in the load-to-failure test by using crowns with clinically relevant curved finish line, dies made of epoxy resin and the rubber sheet. Thus, in the present study, the curved finish line was prepared 0.5 mm above the cement-enamel junction of the anatomic tooth model, and the composite resin-based material (Lava Ultimate) was used as a die material. As such, the discrepancy between their study and ours might be due to the difference in the materials and forms of the crowns. Øilo et al. (33) used alumina-based ceramic crowns with a slight concave occlusal surface without fissures or grooves whereas the crowns tested

in the present study had anatomic form. The test condition, therefore, might cause wedge effect generating tensile stress in the bottom of the occlusal central groove rather than the hoop stress at the cervical area. Clinically relevant testing condition for load-to-failure test using the crowns with anatomic form should be further studied.

All types of composite crowns used in the present study showed higher fracture load than the bite force reported in previous studies. The mean of maximum bite force in the molar regions have been reported to be in a range of 700-900 N, and even the highest value less than 2000 N (23-26). None of the monolithic crowns tested in this study (LU crowns, EC&B-monolayer crowns and EMP crowns) fractured below the load of 2000 N. Furthermore, since lithium disilicate crowns have been successfully used in the molar region (47-49), composite crowns possessing similar fracture resistance as demonstrated in this study may be applicable in the molar regions. However, it should be noted that the load-to-failure test with single loading does not necessarily reflect clinical situations though the test was performed according to the recommendation for a clinically relevant preclinical test (28, 29). It is also known that the fracture resistance of composite crowns is influenced by fatigue procedures, such as cyclic loading, thermal cycling and the combinations (50, 51). Therefore, further studies are necessary to conclude if CAD/CAM-generated composite crowns as well as manually built-up ones can have sufficient fracture resistance after fatigue. Within the limitation of this in vitro study, it is suggested that the composite crowns, especially CAD/CAM-generated crowns may be used in the molar regions because of their supposed sufficient fracture resistance.

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#### References

- 1. DE BACKER H, VAN MAELE G, DE MOOR N, VAN DEN BERGHE L, DE BOEVER J. An 18-year retrospective survival study of full crowns with or without posts. *Int J Prosthodont* 2006; **19**: 136-142.
- 2. PJETURSSON BE, SAILER I, ZWAHLEN M, HAMMERLE CH. A systematic review of the survival and complication rates of all-ceramic and metal-ceramic reconstructions after an observation period of at least 3 years. Part I: Single crowns. *Clin Oral Implants Res* 2007; **18** Suppl 3: 73-85.
- 3. TAKEICHI T, MIURA S, KASAHARA S, EGUSA H, HARA M, SATO T, YOSHINARI M, ODATSU T, WATANABE I, SAWASE T. Update zirconia restorations. *J Proshodont Res* 2014; **Epub ahead of print**.
- 4. LI RW, CHOW TW, MATINLINNA JP. Ceramic dental biomaterials and CAD/CAM technology: State of the art. *J Proshodont Res* 2014; **Epub ahead of print**.
- 5. MIYAZAKI T, NAKAMURA T, MATSUMURA H, BAN S, KOBAYASHI T. Current status of zirconia restoration. *J Proshodont Res* 2013; **57**: 236-261.
- 6. GEURTSEN W. Biocompatibility of dental casting alloys. *Crit Rev Oral Biol Med* 2002; **13**: 71-84.
- 7. WATAHA JC. Biocompatibility of dental casting alloys: A review. *J Prosthet Dent* 2000; **83**: 223-234.
- 8. RAMMELSBERG P, EICKEMEYER G, ERDELT K, POSPIECH P. Fracture resistance of posterior metal-free polymer crowns. *J Prosthet Dent* 2000; **84**: 303-308.
- 9. NAKAMURA T, IMANISHI A, KASHIMA H, OHYAMA T, ISHIGAKI S. Stress analysis of 20

metal-free polymer crowns using the three-dimensional finite element method. Int J Prosthodont 2001; **14**: 401-405.

- 10. BEHR M, ROSENTRITT M, SIKORA MI, KARL P, HANDEL G. Marginal adaptation and fracture resistance of adhesively luted glass fibre-composite reinforced molar crowns with different inner crown surfaces. *J Dent* 2003; **31**: 503-508.
- 11. LEHMANN F, EICKEMEYER G, RAMMELSBERG P. Fracture resistance of metal-free composite crowns–effects of fiber reinforcement, thermal cycling, and cementation technique. *J Proshtet Dent* 2004; **92**: 258-264.
- 12. BEHR M, ROSENTRITT M, HANDEL G. Fiber-reinforced composite crowns and FPDs: A clinical report. *Int J Prosthodont* 2003; **16**: 239-243.
- 13. RAMMELSBERG P, SPIEGL K, EICKEMEYER G, SCHMITTER M. Clinical performance of metal-free polymer crowns after 3 years in service. *J Dent* 2005; **33**: 517-523.
- 14. VANOORBEEK S, VANDAMME K, LIJNEN I, NAERT I. Computer-aided designed/computer-assisted manufactured composite resin versus ceramic single-tooth restorations: A 3-year clinical study. *Int J Prosthodont* 2010; **23**: 223-230.
- 15. FERRACANE JL. Current trends in dental composites. *Crit Rev Oral Biol Med* 1995; **6**: 302-318.
- 16. OGDEN AR. Porosity in composite resins--an Achilles' heel? *J Dent* 1985; **13**: 331-340.
- 17. McCabe JF, Ogden AR. The relationship between porosity, compressive fatigue limit and wear in composite resin restorative materials. *Dent Mater* 1987; **3**: 9-12.
- 18. CHADWICK RG, MCCABE JF, WALLS AW, STORER R. The effect of placement technique upon the compressive strength and porosity of a composite resin. *J Dent* 1989; **17**: 230-233.

- 19. FANO V, ORTALLI I, POZELA K. Porosity in composite resins. *Biomaterials* 1995; **16**: 1291-1295.
- 20. POTICNY D, KLIM J. CAD/CAM in-office technology: innovations after 25 years for predictable, esthetic outcomes. *J Am Dent Assoc* 2010; **141 Suppl 2**: 5S-9S.
- 21. ALT V, HANNIG M, WOSTMANN B, BALKENHOL M. Fracture strength of temporary fixed partial dentures: CAD/CAM versus directly fabricated restorations. *Dent Mater* 2011; 27: 339-347.
- 22. BEHR M, ROSENTRITT M, LATZEL D, KREISLER T. Comparison of three types of fiber-reinforced composite molar crowns on their fracture resistance and marginal adaptation. *J Dent* 2001; **29**: 187-196.
- 23. WALTIMO A, KONONEN M. A novel bite force recorder and maximal isometric bite force values for healthy young adults. *Scand J Dent Res* 1993; **101**: 171-175.
- 24. WALTIMO A, KÖNÖNEN M. Maximal bite force and its association with signs and symptoms of craniomandibular disorders in young Finnish non-patients. *Acta Odontol Scand* 1995; **53**: 254-258.
- 25. WALTIMO A, NYSTRÖM M, KÖNÖNEN M. Bite force and dentofacial morphology in men with severe dental attrition. *Scand J Dent Res* 1994; **102**: 92-96.
- 26. Braun S, Bantleon HP, Hnat WP, Freudenthaler JW, Marcotte MR, Johnson BE. A study of bite force, part 1: Relationship to various physical characteristics. *Angle Orthod* 1995; **65**: 367-372.
- 27. OHLMANN B, GRUBER R, EICKEMEYER G, RAMMELSBERG P. Optimizing preparation design for metal-free composite resin crowns. *J Prosthet Dent* 2008; **100**: 211-219.

- 28. ISO10477. Dentistry Polymer-based crown and bridge materials. 2004.
- 29. ISO6872. Dentistry Ceramic materials. 2008.
- 30. ASTM Standard C 1421-10: Standard test methods for determination of fracture toughness of advanced ceramics at ambient temperature. 2011.
- 31. NAKAMURA K, HARADA A, INAGAKI R, KANNO T, NIWANO Y, MILLEDING P, ÖRTENGREN U. Fracture resistance of monolithic zirconia molar crowns with reduced thickness. *Acta Odontol Scand* 2014; **submitted**.
- 32. PALLIS K, GRIGGS JA, WOODY RD, GUILLEN GE, MILLER AW. Fracture resistance of three all-ceramic restorative systems for posterior applications. *J Prosthet Dent* 2004; **91**: 561-569.
- 33. ØILO M, KVAM K, TIBBALLS JE, GJERDET NR. Clinically relevant fracture testing of all-ceramic crowns. *Dent Mater* 2013; **29**: 815-823.
- 34. Kelly JR. Clinically relevant approach to failure testing of all-ceramic restorations. *J Prosthet Dent* 1999; **81**: 652-661.
- 35. SCHERRER SS, DE RIJK WG. The fracture resistance of all-ceramic crowns on supporting structures with different elastic moduli. *Int J Prosthodont* 1993; **6**: 462-467.
- 36. YUCEL MT, YONDEM I, AYKENT F, ERASLAN O. Influence of the supporting die structures on the fracture strength of all-ceramic materials. *Clin Oral Investig* 2012; **16**: 1105-1110.
- 37. KELLY JR, TESK JA, SORENSEN JA. Failure of all-ceramic fixed partial dentures in vitro and in vivo: analysis and modeling. *J Dent Res* 1995; **74**: 1253-1258.
- 38. YILMAZ H, AYDIN C, GUL BE. Flexural strength and fracture toughness of dental core ceramics. *J Prosthet Dent* 2007; **98**: 120-128.
- 39. WEN MY, MUELLER HJ, CHAI J, WOZNIAK WT. Comparative mechanical property 23

characterization of 3 all-ceramic core materials. Int J Prosthodont 1999; 12: 534-541.

- 40. MORMANN WH, BINDL A, LUTHY H, RATHKE A. Effects of preparation and luting system on all-ceramic computer-generated crowns. *Int J Prosthodont* 1998; **11**: 333-339.
- 41. SAKOGUCHI K, MINAMI H, SUZUKI S, TANAKA T. Evaluation of fracture resistance of indirect composite resin crowns by cyclic impact test: Influence of crown and abutment materials. *Dent Mater J* 2013; **32**: 433-440.
- 42. ROSENTRITT M, PLEIN T, KOLBECK C, BEHR M, HANDEL G. In vitro fracture force and marginal adaptation of ceramic crowns fixed on natural and artificial teeth. *Int J Prosthodont* 2000; **13**: 387-391.
- 43. QUINN JB, QUINN GD, KELLY JR, SCHERRER SS. Fractographic analyses of three ceramic whole crown restoration failures. *Dent Mater* 2005; **21**: 920-929.
- 44. SCHERRER SS, QUINN GD, QUINN JB. Fractographic failure analysis of a Procera AllCeram crown using stereo and scanning electron microscopy. *Dent Mater* 2008; **24**: 1107-1113.
- 45. ØILO M, GJERDET NR. Fractographic analyses of all-ceramic crowns: A study of 27 clinically fractured crowns. *Dent Mater* 2013; **29**: e78-84.
- 46. ØILO M, HARDANG AD, ULSUND AH, GJERDET NR. Fractographic features of glass-ceramic and zirconia-based dental restorations fractured during clinical function. *Eur J Oral Sci* 2014; **122**: 238-244.
- 47. REICH S, SCHIERZ O. Chair-side generated posterior lithium disilicate crowns after 4 years. *Clin Oral Investig* 2013; **17**: 1765-1772.
- 48. ESQUIVEL-UPSHAW J, ROSE W, OLIVEIRA E, YANG M, CLARK AE, ANUSAVICE K. 24

Randomized, controlled clinical trial of bilayer ceramic and metal-ceramic crown performance. *J Prosthodont* 2013; **22**: 166-173.

- 49. PIEGER S, SALMAN A, BIDRA AS. Clinical outcomes of lithium disilicate single crowns and partial fixed dental prostheses: A systematic review. *J Prosthet Dent* 2014; **112**: 22-30.
- 50. ATTIA A, ABDELAZIZ KM, FREITAG S, KERN M. Fracture load of composite resin and feldspathic all-ceramic CAD/CAM crowns. *J Prosthet Dent* 2006; **95**: 117-123.
- 51. KAWANO F, OHGURI T, ICHIKAWA T, MATSUMOTO N. Influence of thermal cycles in water on flexural strength of laboratory-processed composite resin. *J Oral Rehabil* 2001; **28**: 703-707.

# **Tables**

Table 1. Materials used in this study

|                 |                 |         | Filler                                |         |                   |  |  |
|-----------------|-----------------|---------|---------------------------------------|---------|-------------------|--|--|
| Product name    |                 | Code    | Composition                           | content | Manufacturer      |  |  |
|                 |                 |         |                                       | (wt.%)  |                   |  |  |
| Lava Ultimate   |                 | LU      | Bis-GMA, Bis-EMA, UDMA,               |         | 3M/ESPE,          |  |  |
|                 |                 |         | TEGDMA, silica particles (20 nm),     | 00      | St. Paul,         |  |  |
|                 |                 |         | zirconia particles (4-11 nm),         | 80      | MN,               |  |  |
|                 |                 |         | Nanoparticle clusters (0.6-10 μm)     |         | USA               |  |  |
| Estenia<br>C&B  | Opacious Dentin | EC&B-OD | UDMA, UTMA, Bis-GMA,                  |         | Kuraray/Noritake, |  |  |
|                 | Dentin          | EC&B-D  | alumino-silicate glass particles (1.5 | 92      | Tokyo,            |  |  |
|                 | Enamel          | EC&B-E  | μm), alumina particles (20 nm)        |         | Japan             |  |  |
|                 |                 |         |                                       |         | Ivoclar/Vivadent, |  |  |
| IPS e.max press |                 | EMP     | lithium disilicate glass-ceramic      | -       | Schaan,           |  |  |
|                 |                 |         |                                       |         | Liechtenstein     |  |  |

Table 2. Number and total volume of voids inside the crowns.

|                       | Number of voids |      |            |   | Volume of voids (mm <sup>3</sup> ) |        |            |  |
|-----------------------|-----------------|------|------------|---|------------------------------------|--------|------------|--|
|                       | Mean            | SD   | Statistics | 1 | Mean                               | SD     | Statistics |  |
| LU crowns             | N.D.            | N.D. | а          |   | N.D.                               | N.D.   | a          |  |
| EC&B-layered crowns   | 46.5            | 18.7 | b          |   | 0.0206                             | 0.0171 | b          |  |
| EC&B-monolayer crowns | 0.7             | 0.8  | а          |   | 0.0001                             | 0.0001 | а          |  |
| EMP crowns            | 61.3            | 15.4 | b          |   | 0.0009                             | 0.0003 | С          |  |

Statistical significance is expressed between the different letters (p<0.05). N.D.: not detected.

#### Figure legends

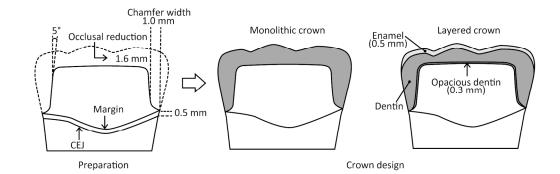
Fig. 1. Schematic of the preparation and the crown designs for monolithic crowns (Lava Ultimate crowns, Estenia C&B -monolayer crowns and IPS e.max press crowns) and layered crown (Estenia C&B -layered crowns). CEJ: cement-enamel junction of the tooth model.

Fig. 2 Comparison of (a) flexural strength, (b) modulus of elasticity and (c) fracture toughness between the materials used in this study. Each value represents the mean of ten measurements for (a) and (b), and five measurements for (c) with standard deviation. Different letters above the columns show significant differences (p<0.01). LU: Lava Ultimate, EC&B: Estenia C&B, OD: opacious dentin, D: dentin, E: enamel, EMP: IPS e.max press.

Fig. 3. Representative micro-CT images of the crowns tested. White arrows indicate the existence of voids. LU: Lava Ultimate, EC&B: Estenia C&B, EMP: IPS e.max press.

Fig. 4. Fracture load of the crowns. Each value represents the mean of six measurements with standard deviation. \*\*: p<0.01, \*: p<0.05. LU: Lava Ultimate, EC&B: Estenia C&B, EMP: IPS e.max press.

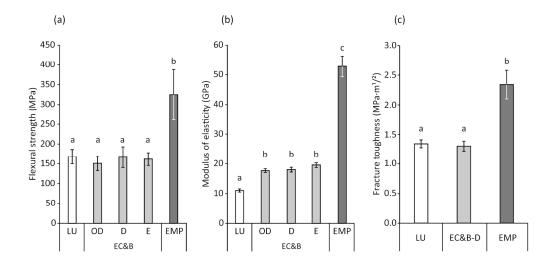
Fig. 5. Representative SEM composite views of fracture surface of (a) Lava Ultimate crown, (b) Estenia C&B-layered crown, (c) Estenia C&B-monolayer crown, and (d) IPS e.max press crown. Each image was created by overlapping 11-13 original SEM images photographed at ×40. Large and small arrows display the fracture origin and voids, respectively.



Schematic of the preparation and the crown designs for monolithic crowns (Lava Ultimate crowns, Estenia C&B -monolayer crowns and IPS e.max press crowns) and layered crown (Estenia C&B -layered crowns).

CEJ: cement-enamel junction of the tooth model.

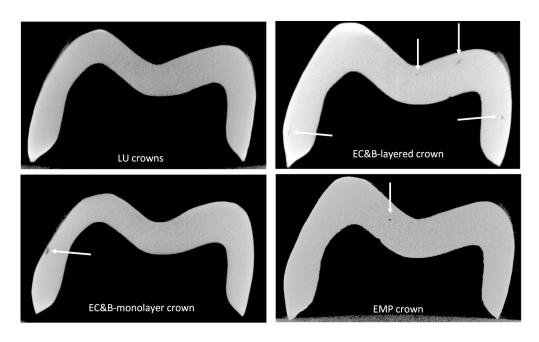
167x55mm (300 x 300 DPI)



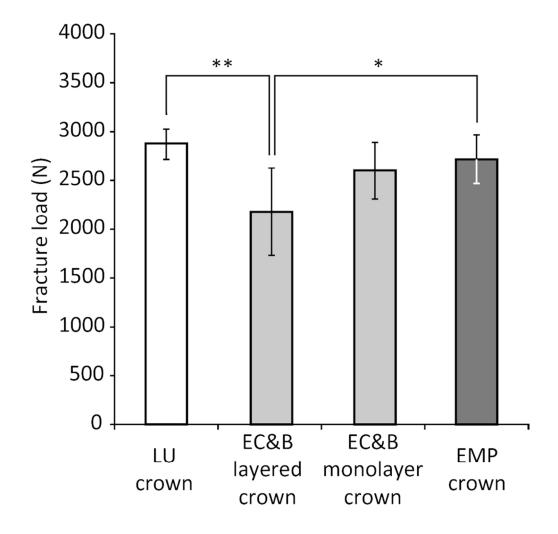
Comparison of (a) flexural strength, (b) modulus of elasticity and (c) fracture toughness between the materials used in this study. Each value represents the mean of ten measurements for (a) and (b), and five measurements for (c) with standard deviation. Different letters above the columns show significant differences (p<0.01). LU: Lava Ultimate, EC&B: Estenia C&B, OD: opacious dentin, D: dentin, E: enamel, EMP: IPS e.max press.

162x77mm (300 x 300 DPI)

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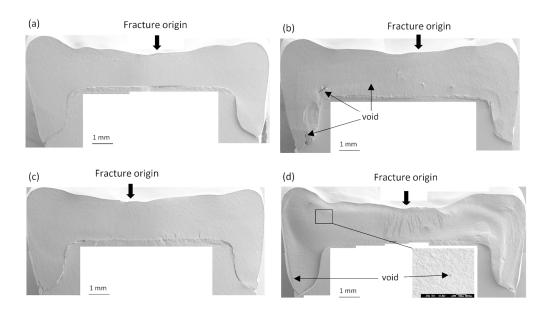
Representative micro-CT images of the crowns tested. White arrows indicate the existence of voids. LU: Lava Ultimate, EC&B: Estenia C&B, EMP: IPS e.max press.  $161 \times 97 \text{mm} \; (300 \times 300 \; \text{DPI})$ 



Fracture load of the crowns. Each value represents the mean of six measurements with standard deviation.

\*\*: p<0.01, \*: p<0.05. LU: Lava Ultimate, EC&B: Estenia C&B, EMP: IPS e.max press.

77x75mm (300 x 300 DPI)



Representative SEM composite views of fracture surface of (a) Lava Ultimate crown, (b) Estenia C&B-layered crown, (c) Estenia C&B-monolayer crown, and (d) IPS e.max press crown. Each image was created by overlapping 11-13 original SEM images photographed at ×40. Large and small arrows display the fracture origin and voids, respectively.

164x91mm (300 x 300 DPI)