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Comparing Marginal Gap and Flexural Strength Between Additive and Subtractive Manufactured Provisional and Permanent Crown Materials

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Full title: Comparing Marginal Gap and Flexural Strength Between Additive and Subtractive Manufactured Provisional and Permanent Crown Materials

Working title: A comparison of additive vs. subtractive manufacturing techniques when assessing flexural strength and marginal accuracy

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ABSTRACT

Purpose: The purpose of this study was to compare the flexural strength, Weibull reliability, marginal gap, and internal discrepancy amongst different types of provisional and permanent crown materials fabricated using additive and subtractive manufacturing processes.

Materials and Methods: IPS e.max CAD (EC) and Paradigm MZ100 (PM) for subtractive manufacturing, and FormLabs Permanent Crown (PCR) and Temporary CB (TCR) resins for additive manufacturing were investigated. Flexural strength was tested using sectioned bars (1.3 x 4 x 18 mm) for EC and PM, and printed bars for PCR and TCR (n = 15 per material). The EC bars were crystallized. All bars were polished and subjected to a 3-point bend test using a universal testing machine (Instron E3000). Additionally, a mandibular left first molar typodont tooth was prepared, optically scanned, designed, and crowns were milled (in EC and PM) and 3D-printed (in PCR and TCR) (n = 5 per material). Marginal gap and internal discrepancy were measured using micro-computed tomography (SkyScan 1172). Weibull modulus and characteristic strength were computed per group. Flexural strength and marginal discrepancy data was analyzed with t-test and compared using one-way ANOVA with Tukey HSD post-hoc comparisons ($\alpha = 0.05$).

Results: Flexural strengths were significantly different between sectioned (EC and PM) and printed (PCR and TCR) bars. Mean flexural strength (MPa) in descending order were EC = 335.50 ± 28.97, PM = 154.34 ± 21.03, PCR = 128.09 ± 7.30, and TCR = 126.29 ± 9.23. One-way ANOVA revealed significant differences amongst EC, PM, and the 3-D printed groups ($F = 426.460$, $p < 0.001$). No statistically significant difference in flexural strength was observed between PCR and TCR. For the Weibull modulus, PCR (21.07) presented with the highest modulus followed by EC (13.70), TCR (16.52). PM (8.77) resulted in the lowest modulus, indicating its poor reliability. For marginal gap comparison, EC and PCR showed significantly lower marginal gaps than PM and TCR (F = 25.083, $p < 0.001$). PM had the smallest internal margin discrepancy. Internal margin discrepancies of 3-D printed crowns were higher than milled crowns, but there was no statistically significant difference in marginal gaps between EC, PM, and PCR.

Conclusion: Within the limitation of this in vitro study, the resin material sectioned from milled blocks exhibited higher flexural strength than those fabricated with 3-D printed materials. There was no significant difference in flexural strength between the 3-D printed permanent and temporary crown materials tested in this study. The marginal gap analysis was varied with milled EC having the smallest clinical margin and PM having the smallest internal gap. Crowns fabricated with 3-D printing (PCR) had the best overall marginal reproduction, but EC was the only material tested that did not have a marginal gap above 120 microns.

Keywords. Flexural Strength, Marginal Gap, Additive and Subtractive Manufacturing, 3-D Printing, Ceramics, Crown resin

Introduction

The use of additive manufacturing (AM) or 3-D printing has gained popularity in an ever-increasing variety of industries, including dentistry. Additive manufacturing is defined by the American Society for Testing and Materials (ASTM) as the process of joining materials to make objects from 3D model data, usually layer upon layer, as opposed to subtractive manufacturing methodologies.[1] According to the International Standards Organization (ISO)[2], there are seven general types of AM: material extrusion, vat polymerization, powder bed fusion, material jetting, binder jetting, direct energy deposition, and sheet lamination. The benefits of AM compared to subtractive manufacturing (SM) are less material waste, reduced cost, and the ability to manufacture intricate details and complex geometries.[3] The limitations of AM are: initial start-up cost can be expensive, a limited range of dental materials are available for printing, and post-processing can add time and cost to the overall operation. For dentistry, one major disadvantage of AM is its inability to print ceramics and metals in room temperature.

For over 20 years, SM is a relatively mature technology that has been widely used for the fabrication of ceramic restorations. Two distinctive advantages of SM are: a wide selection of materials is available for milling, and SM prototypes require less post-processing treatment to produce a higher degree of surface finish than AM. Nevertheless, there are some inherent drawbacks associated with this technology. Examples include the risk of microcrack introduction during milling, the need for worn tool replacement, generation of material waste, and limitations in reproducing surface geometry as dictated by the size of the milling tools and the working axes of the computer numerical control machine.[1,4] Often, dentists leverage the advantages of both AM and SM, tailoring their approach to meet the unique needs of each patient.

Recently, two novel 3-D printing Permanent Crown and Temporary Crown resins, were developed by Bego (GmbH & Co., Bremen, Germany), and introduced by Formlabs (Somerville, MA, USA). Bego called these resins, VareoSmile Crown Plus and VareoSmile Temp, respectively. The manufacturer claimed they are the first tooth-colored, ceramic-filled resins for 3-D printing. However, research comparing the strength, reliability, and accuracy of these new materials with existing milling materials in the market are sparse.

The success of a fixed prosthesis is partially dependent on the accuracy of its fit and the mechanical properties of the material. For this study, accuracy of the manufacturing process as well as crown material was evaluated via marginal and internal gap assessment. Most studies that evaluated internal or clinical accuracy used either loupes or a microscope to assess the marginal gap.[5] For a nondestructive assessment, this study utilized a micro-computed tomography (micro-CT) for volumetric and 3-D investigations of crown's internal gaps.

The materials being used for this study, with exception to lithium disilicate, are primarily composite resins with different filler content. The 3M MZ100 product is composed of Bisphenol A diglycidyl ether combined with a methacrylic acid to produce the diester Bis-GMA (bisphenol A-glycidyl methacrylate). Bis-GMA is responsible for the resin's viscosity.[6] The other main component of MZ100 are the ultrafine zirconia-silica ceramic particles, from which these fillers enhance strength, hardness, and wear resistance.[7] The Formlabs resin materials, both permanent and temporary, are composed of 4,4'-isopropylidenediphenol or "BPA", which is primarily used as a co-monomer in the production of polycarbonates.[8] 2-Methylprop-2-enoic acid, also known as methacrylic acid, is widely used in the production of polymers, resins, and coatings, as well as, in the production of dental materials and adhesives.[9] Also, silanized "dental glasses" were added for strength and esthetics, and diphenyl(2,4,6-trimethylbenzoyl) phosphine oxides were added as photoinitiators.[10,11]

The purpose of this study was to: 1) Evaluate and compare the flexural strength of two milled and two 3-D printed crown materials, and 2) Evaluate and compare marginal fit of milled lithium disilicate crowns, milled composite resin crowns, and 3-D printed permanent and temporary resin crowns using microcomputed tomography. The postulated null hypothesis was there would be no difference in marginal adaptability and flexural strength between the milled and 3-D printed restorations.

Materials and Methods

Four different materials and two different manufacturing methods for crown fabrication were compared: (1) IPS e.max CAD (EC – Ivoclar Vivadent, Schaan, Liechtenstein) and Paradigm MZ100 (PM – 3M, Saint Paul, MN) for milling, and (2) Permanent Crown Resin (PCR – FormLabs, Somerville, MA) and Temporary CB Resin (TCR – FormLabs, Somerville, MA) for 3D-printing (Table 1). The 3-D printed materials were manufactured by BEGO GmbH & Co out of Bremen, Germany, but sold and distributed through FormLabs.[12]

A total of 80 specimens were tested: 15 bars for each material for flexural strength testing without aging, and 5 crowns for each material for marginal gap analysis. The effective size was estimated from previous studies in the literature. The required number of samples needed to satisfy a one-way ANOVA, with a type I error of 0.05 and a power of 80% for detecting a 15% difference in strength and marginal discrepancy, was 15 per group for the strength test and 5 per group for the marginal gap test.

Flexural Strength Testing

To evaluate TCR and PCR materials, a rectangular bar (1.3 mm x 4 mm x 18 mm) was digitally designed using a computer-aided design software (Autodesk Meshmixer, Autodesk Inc., San Rafael, CA) and exported in standard tessellation language (STL) format. A 3-D printer (Formlab2, FormLabs, Somerville, MA) was used to print TCR and PCR bars (n = 15 per material). The 3-D printed bars were fabricated using a STL file. STLs were loaded into a slicing software (PreForm 3.21.0, FormLabs, Somerville, MA), then printed on a stainless-steel build platform. Supports were automatically generated, and any supports located on or near the tested surface were repositioned. (Figure 1) Once the bars were manufactured, they were placed in a resin wash station (Form Wash, FormLabs, Somerville, MA) filled with isopropyl alcohol (IPA, 70%) and washed for 3 minutes. Any excess resin was removed with a brush. The bars were first dried using compressed air until the surface appeared dry and lacked residual shine, then left to air dry for 30 minutes. To cure the samples, the raft (with supports still intact) was placed in the resin curing station (Form Cure, FormLabs, Somerville, MA) with the raft side down and cured at 60°C for 20 minutes. A handpiece with a cutting disc was used to separate supports and the raft from the printed bars. The bars were then particle abraded with glass beads to remove residual coating at a maximum pressure of 1.5 bar for 10 seconds. Next, the crowns were placed back in the curing station and post-cured again at 60 °C for an additional 20 minutes.[3] To evaluate EC and PM materials, blocks were sectioned into rectangular bars (1.3 mm x 4 mm x 18 mm) using a low-speed precision cutter (Isomet Saw, Buehler, Lake Bluff, IL). All specimens were polished with 320 and 600 grit SiC abrasive paper (3M ESPE, Saint Paul, MN). The EC bars were fired in a ceramic furnace (Programat P310, Ivoclar Vivadent, Schaan, Liechtenstein) following manufacturer's instructions.[13]

Flexural strength testing was completed in accordance with the international standard for ceramic materials ISO 6872:2015.[14] All specimens were tested in a universal testing machine (ElectroPuls 3000, Instron, Norwood, MA) with a crosshead speed of 1 mm/min, and data was recorded using a proprietary software (Bluehill, Instron, Norwood, MA).[15] The flexural strength was obtained using the equation FS = $3Fl/(2bd^2)$, where F was the loading force at the fracture point, l was the length of the support span (15 mm), b was the width of the beam specimen and d was the depth. Measurements for b and d were made using electronic digital calipers (GA182, Grobet Vigor, Carlstadt, NJ).

Weibull Statistical Analysis

Fifteen specimens were used for each group based on the ISO 6872:2015. The Weibull's 2-parameter (σ_0 and m) distribution function is:

$$P_f = 1 - e^{-\left(\frac{\sigma}{\sigma_0}\right)^m} \qquad \textit{(1)}$$

where P_f is the cumulative probability of failure, σ [MPa] is the flexural strength, σ_0 [MPa] is the Weibull characteristic strength, and m is the Weibull modulus.

The two parameters, σ_0 and m, were calculated by using rank order statistics. For example, after the flexural strengths for all 15 specimens were measured, the flexural strength (σ) values were ranked in ascending order and were assigned a probability of failure per each specimen based on its ranking using the following equation:

$$P_f(\sigma_i) = \frac{i - 0.5}{N} \qquad \textit{(2)}$$

where *i* represents 1, 2, 3, …, N of the i^{th} datum in ascending order of the flexural strength (σ_i), and N is the total number of specimens tested. Next, equation *(1)* was rearranged into the following equation:

$$ln\left[ln\left(\frac{1}{1-P_f}\right)\right] = m\ ln\sigma_i - m\ ln\sigma_0 \qquad (3)$$

Then, $ln\left[ln\left(\frac{1}{1-P_f}\right)\right]$ was plotted on the ordinate against $ln(\sigma_i)$ on the abscissa, from which a maximum likelihood estimation procedure was used to estimate the linear regression model. The slope and intercept of equation *(3)* yield m and $m\ ln\sigma_0$ respectively. Then, σ_0 was solved from the intercept. When the characteristic strength is the strength value, the cumulative probability of failure, P_f, was at 63.2%.

Marginal Gap Testing

A mandibular left first molar typodont tooth was mounted with its adjacent teeth on a typodont and prepared for a full coverage lithium disilicate crown following the manufacturer's guidelines (shoulder/chamfer 1.0 mm and occlusal reduction 1.5 mm).[16] After tooth preparation, the preparation was scanned using a chairside intra-oral scanner (CEREC Primescan, Dentsply Sirona, Charlotte, North Carolina). The prepared tooth was scanned, and the crown template was digitally designed with CAD software (inLab version 22.1, Dentsply Sirona, Charlotte, North Carolina) by one comprehensive dentistry resident. Crowns were milled (EC and PM) using a 4-axis mill (CEREC MC XL, Dentsply Sirona, Charlotte, North Carolina). A new set of burs was installed prior to milling each material group. The EC crowns were subjected to crystallization firing procedure per the manufacturer's instructions using a ceramic furnace (Programat P310, Ivoclar Vivadent, Schaan, Liechtenstein).[17]

The 3-D printed crowns were fabricated using the same STL file used for the milled crowns. STLs were loaded into a slicing software (PreForm 3.21.0, FormLabs, Somerville, MA), then printed on a stainless-steel build platform with the occlusal planes facing the build platform. Supports were automatically generated, and any supports located on or near the margins were repositioned. Once the crowns were manufactured, they were placed in a resin wash station (Form Wash, FormLabs, Somerville, MA) filled with isopropyl alcohol (IPA 70%) and washed for 3 minutes. Any excess resin was removed with a brush. The crowns were first dried using compressed air until the surface appeared dry and lacked residual shine, then left to air dry for 30 minutes. (Figure 1) To cure the samples, the raft (with supports still intact) was placed in the resin curing station (Form Cure, FormLabs, Somerville, MA) with the raft side down and cured at 60°C for 20 minutes. A handpiece with a cutting disc was used to separate supports and the raft from the printed crowns. The crowns were then particle abraded with glass beads to remove residual coating at a maximum pressure of 1.5 bar for 10 seconds. Next, the crowns were placed back in the curing station and post-cured again at 60 °C for an additional 20 minutes, with the occlusal plane facing upwards. Lastly, the crowns were polished using fine pumice stone and polishing compound prior to use.[18]

The mean values and standard deviations for marginal fit were captured by group for EC, PM, PCR and TCR. Marginal gaps (µm) were measured utilizing a micro-computed tomography (micro-CT, SKYSCAN 1172, Bruker, Billerica, MA). Five crowns per group were scanned 180 degrees with a 0.4° rotational increment using a source voltage and current of 44 kV and 222 µA with a 0.5 mm Al filter and an 8.31 µm image-pixel size, for a total of 1048 × 2000 pixels per slice. The scanned images were reconstructed into 3-D images using NRecon software (v. 2.0, Bruker, Billerica, MA). Marginal discrepancies (4 surfaces per crown) were measured using CTAn software (v.1.18, Bruker micro-CT). For each surface, five slices were assessed at 100 slices apart, from which two measurements per slice, an internal gap, and a clinical gap were recorded. The internal gap was measured as the closest two points from the internal crown margin to the internal tooth margin. The clinical gap was measured from extrapolation of an imaginary line that follows the crown contour from where the crown margin ended down to the typodont tooth surface (Figures 2a-c).

Results

For flexural strength, EC (335.52 ± 28.97 MPa) displayed the highest strength amongst all materials, followed by PM (154.34 ± 21.03 MPa), PCR (128.09 ± 7.30 MPa), and TCR (126.29 ± 9.23 MPa). One-way ANOVA revealed significant differences between EC, PM, and the 3-D printed groups ($F = 426.460$, $p < 0.001$). PCR was not statistically significantly different from TCR (Figure 3). For the Weibull modulus, PCR (21.07) presented with the highest modulus followed by EC (13.70), TCR (16.52). PM (8.77) resulted in the lowest modulus, indicating its poor reliability (Table 2).

Data for marginal gaps are presented in Figure 4. One-way ANOVA showed that EC (79.97 ± 22.42 µm) and PCR (88.58 ± 35.23 µm) had significantly lower clinical gaps than TCR (105.94 ± 46.79 µm) and PM (119.30 ± 40.42 µm) ($F = 25.083$, $p < 0.001$). PM (14.56 ± 19.04 µm) had the smallest internal margin. PCR (25.01 ± 21.00 µm) and EC (29.88 ± 20.85 µm) were not statically significantly different than each other, but they did have statistically significantly larger gaps than PM. The widest internal margin was TCR (38.60 ± 42.25 µm), but it was not significantly different than EC samples (Figure 4). When looking at the surfaces specifically, the mesial surface (0.079 ± 0.044 µm) had a significantly smaller clinical gap than the distal (0.099 ± 0.035 µm), buccal (0.103 ± 0.035 µm), and lingual (0.111 ± 0.042 µm), with the later three having no statistical difference. As for the internal gap, it was the reverse; the buccal (0.020 ± 0.022 µm), distal (0.021 ± 0.020 µm), and lingual (0.028 ± 0.029 µm) internal margins were not statistically different from each other, but they were all significantly smaller than the mesial (0.039 ± 0.037 µm) internal margin (Figure 5).

Discussion

The flexural strength data as well as the marginal gap data both showed statistical differences between the subtractive and additive manufactured permanent and temporary crown materials being evaluated. Due to these findings, the null hypothesis was rejected. Interestingly, there was a lack of statistical difference in the flexural strengths of permanent and temporary crown resins (PCR and TCR) evaluated in this study. FormLabs currently sells and distributes their crown resin from a separate manufacturer (BEGO), and the company does not advertise or release their proprietary material composition and percentages.[3] Based on the results of this study, the similarity of the two products could mean that a clinician would be able to achieve reasonably similar clinical results in terms of fracture resistance when using either the permanent or temporary material. Clinicians may need to be selective in choosing PCR in high stress bearing areas. Additional testing and research simulating intra-oral environment will be necessary for definitive assessment.

Nguyen et al. found in their study that high temperature/high pressure polymerization resulted in a significant increase in flexural strength, hardness, and density for indirect and direct composites. In this study, multiple indirect and direct composite resins were photo-polymerized at 180 degrees Celsius at a constant pressure of 250 MPa for 60 min. It is within reason to hypothesize that the CAD/CAM blocks manufactured under pressure and high temperatures resulted in a higher flexural strength than materials manufactured by 3D printing. As for Weibull outcomes, the manufacturing process of the CAD/CAM blocks appeared to have a less reliable reproducibility than can be produced with 3D printing.[19]

For marginal gap analysis, the outcome data varied. For internal margin, the subtractive resin (PM) and additive permanent resin (PCR) performed the best with the smallest gaps. For clinical gap, EC and PCR performed the best. Overall, the data seemed to indicate PCR as the most accurate overall, but EC was the only material that did not have a clinical margin above the 50-120 microns recommended by McLean and Von Fraunhofer.[20] When evaluating the accuracy and trueness of additive manufacturing against subtractive manufacturing, Kakinuma et al. found 3-D printed crowns showed higher accuracy with fewer marginal discrepancies than milled crowns. In that study, they also found 3-D printed crowns showed a high trueness value of <50 um in all areas, and the same trend was observed across the different crown designs. Milled crowns showed significant positive deviations on the internal surface of the cusps in all preparations with sharp occlusal-axial line angles. Moreover, 3-D printed crowns showed superior trueness values compared with the milled crowns in all groups of abutment models, whose preparation were designed with rounded occlusal-axial line angles and rounded shoulder margins. Milled crowns showed significant dimensional deviations, especially at the cusps.[21] Kakinuma et al.'s study supports the findings of our study indicating the high accuracy of 3D printing even when the underlying prep has sharper angles.

Some limitations seen in this study include, using 70 percent isopropyl alcohol instead of the manufacturers recommended 90 percent or greater. This could prevent the complete removal of excess resin from within the crown and alter the internal fit of the crown. The points used for measuring both clinical and internal gaps could not be standardized. Due to this variation in points, gaps could be wider or narrower depending on a changing measuring point. Observer fatigue also could have led to varying marginal and internal gaps. One observer looking at multiple slices over multiple hours a day, affected the ability to reselect similar points each time.

Conclusions

Within the limitation of this in vitro study, the resin materials sectioned from milled blocks exhibited higher flexural strength than those fabricated with 3-D printed materials. There was no significant difference in flexural strength between the 3-D printed permanent and temporary crown material tested in this study. The marginal gap analysis was varied with milled EC having the smallest clinical margin and PM having the smallest internal gap. Crowns fabricated with 3-D printing (PCR) had the best overall marginal reproduction, but EC was the only material tested that did not have a gap above 120 microns.

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Table 1 Investigated Materials used for crown restorations

Product	Manufacturer	Color	Composition
IPS e.max CAD (EC)	Ivoclar Vivadent AG	A2	SiO2 (57-80% by weight), Li2O (11-19% by weight), K2O (0-13% by weight), P2O5 (0-11% by weight), ZrO2 (0-8 by weight), ZnO (0-8% by weight), Al2O3 (0-5 by weight), MgO (0-5% by weight), Colouring Oxides (0-8% by weight)[13]
Paradigm MZ100 (PM)	3M	A2	85 wt% ultrafine zirconia-silica ceramic particles, Bisphenol A diglycidyl ether dimethacrylate, tri[ethyleneglycol] dimethacrylate[7]
Permanent Crown (PCR)	Formlabs GmbH	A2	Esterification products of 4,4'-isopropylidenediphenol, ethoxylated and 2-methylprop-2-enoic acid (50-75 % Weight) Silanized dental glass, Diphenyl(2,4,6-trimethylbenzoyl) phosphine oxide (<3% by weight)[10, 11]
Temporary CB (TCR)	Formlabs GmbH	A2	Esterification products of 4,4'-isopropylidenediphenol, ethoxylated and 2-methylprop-2-enoic acid (50-75 % weight) Silanized dental glass, diphenyl(2,4,6-trimethylbenzoyl) phosphine oxide (< 2.5 by weight)[10, 11]

Table 2 Comparison of the reliability parameters (e.g., Weibull modulus) amongst the four different materials

Material	Weibull Modulus	Characteristic Strength (MPa)
e.max CAD (EC)	13.70	348.28
Paradigm MZ100 (PM)	8.77	163.06
Permanent Crown (PCR)	21.07	131.33
Temporary CB (TCR)	16.52	130.30

Figure 1 3-D pritned PCR crown and bar samples

Figure 2a Indication of how measurements were taken for internal and clinical gap at 910 magnifications

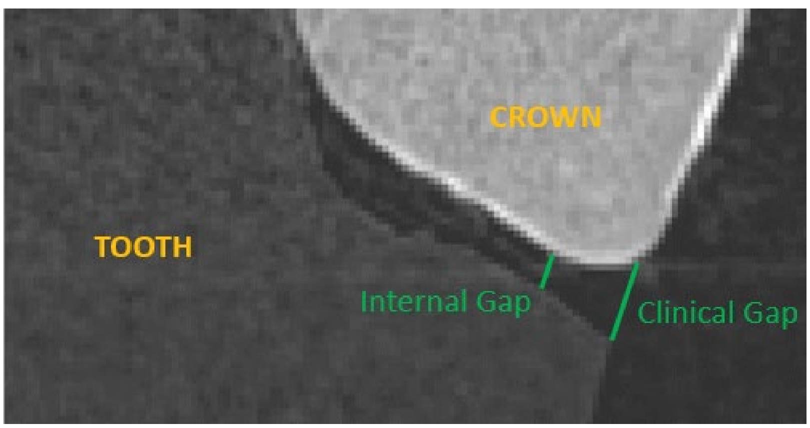

Figure 2b Micro-CT before magnification

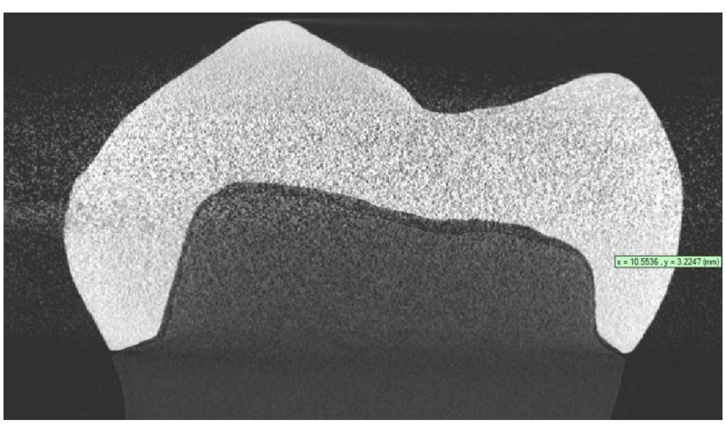

Figure 2c Micro-CT at x 910 magnification

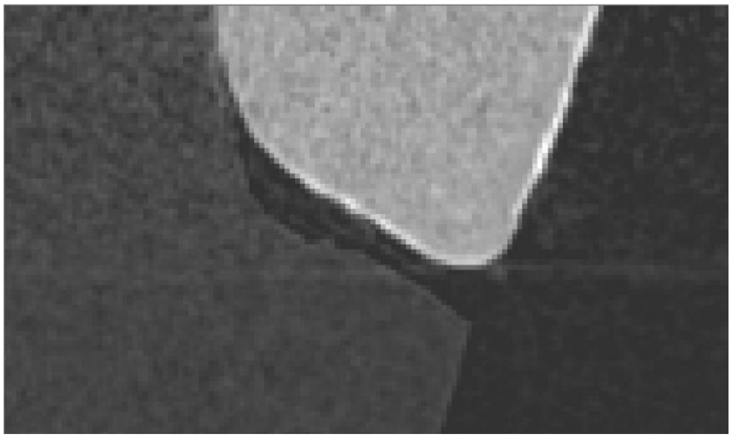

Figure 3 Flexural strength comparison amongst material types is plotted. Materials with the same letter are not statically significant from each other ($p > 0.05$). Bars in green represent subtractive material and bars in blue indicate additive materials.

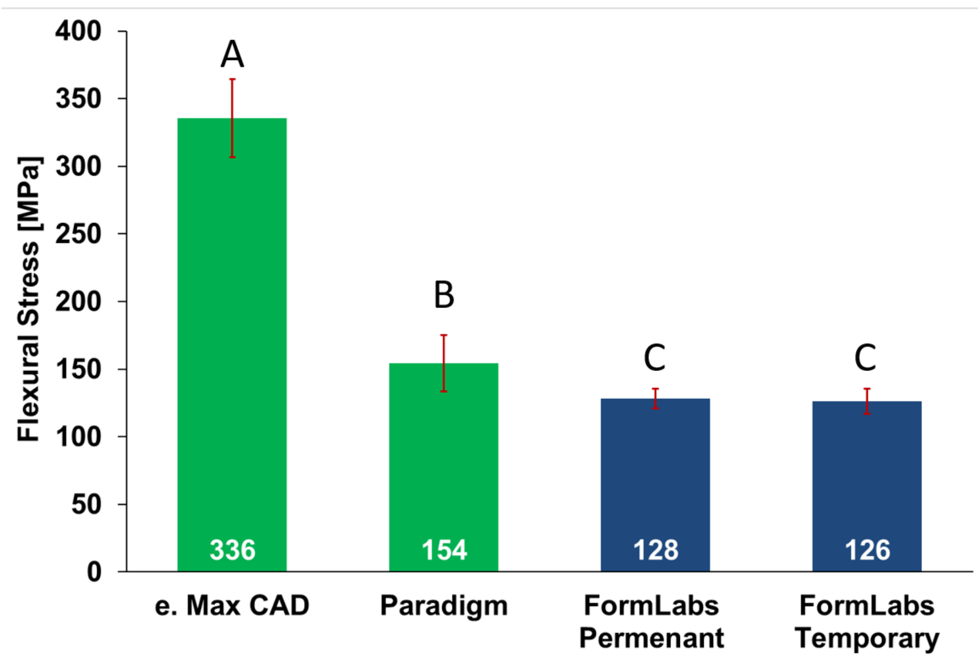

Figure 4 Comparison of marginal discrepancies amongst various crowns fabricated from four different materials is shown. Materials with the same case letter are not statistically significant from each other ($p > 0.05$). Bars in green represent subtractive material and bars in blue indicate additive materials.

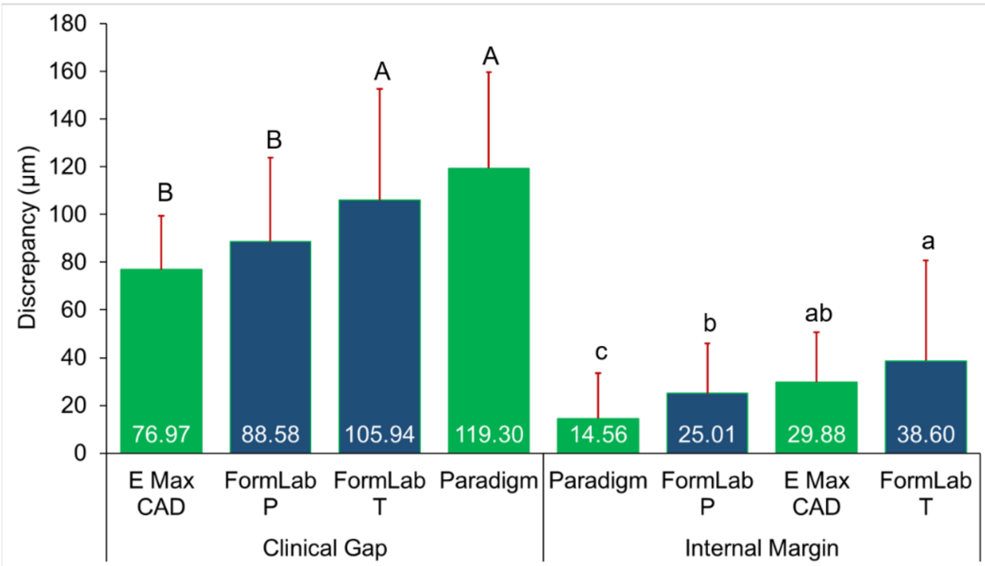

Figure 5 Comparison of discrepancies amongst mesial, distal, buccal, and lingual crown surfaces is shown. Materials with the same case letter are not statically significant from each other ($p > 0.05$). The orange color scale represents each individual surface from dark orange (mesial) to lightest shade of orange (lingual).