

The Effect of Cyclic Loading on Fracture Strength of 3D Printed Zirconia Crowns and CAD/CAM Zirconia Crowns

An In-Vitro Study

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List of abbreviations

AG	Axial gap
ANOVA	Analysis of variance
CG	Cervical gap
FDM	Fused deposition modeling
IJP	Ink-jet printing
LCM	Lithography-based Ceramic Manufacturing
MG	Marginal gap
OG	Occlusal gap
SEM	Scanning electron microscope
SLA	Stereo-lithography
SLM	Selective laser melting
SLS	Selective laser sintering
SRT	Silicone replica technique
VMGT	Vertical marginal gap technique
Y-TZP	Yttria partially stabilized zirconia

1. English summary

1.1 Introduction

For several years, the demand for metal-free tooth-colored restorations has increased because of the increased esthetic demands, more conservative preparations, and the high concerns about toxic and allergic reactions to certain alloys (Erođlu and Gurbulak, 2013). Yttria partially stabilized zirconia (Y-TZP) ceramics have been widely used as a core substructure owing to their biocompatibility, superior esthetics and high fracture resistance (Stawarczyk et al., 2016). Zirconia core can be veneered with glass ceramics to improve esthetics, but the main drawback is the risk of being chipped during function (Kim et al., 2008). The innovation of monolithic zirconia crowns with full anatomical contours eliminates the need for veneering (Choi et al., 2017).

The development of these materials and the fabrication techniques allow the preservation of tooth structure and the usage of various restorations, such as partial coverage restorations, crowns and bridges (Patroni et al., 2010). Zirconia restorations are mainly fabricated using subtractive milling techniques but there is a possibility of thin margins chipping off during milling, the difficulty of creating deep grooves due to the thickness of milling bur tip, the waste material generated during milling and the wear of the milling burs (Alao et al., 2017; Al Hamad et al., 2022; Li et al., 2020; Luthardt et al., 2004).

Innovations in additive manufacturing in the last few years made it possible to use various materials to fabricate dental restorations. Additive fabrication techniques include selective laser sintering (SLS), selective laser melting (SLM), stereo-lithography (SLA), ink-jet printing (IJP), fused deposition modeling (FDM), and others (Tahayeri et al., 2018). In this study, Lithography-based Ceramic Manufacturing (LCM) technology was used for the fabrication of a fully anatomical 3D printed zirconia crown as it is the only available and most advanced approach (Schweiger et al., 2021).

Marginal adaptation of dental crowns is an important factor that may lead to clinical failure (Euán et al., 2012; Limkangwalmongkol et al., 2009). It is defined as the vertical distance between the restoration margin and the finish line of the prepared tooth (Holmes et al., 1989; Quintas et al., 2004). A stereomicroscope can be used to measure the mar-

ginal gap under high magnification (Euán et al., 2014). The clinically acceptable marginal gap is reported to be between 50 and 120 μm (Euán et al., 2012). In addition, the internal fit is an important factor in the success of dental crowns, as it affects the retention of the dental crown, rotational stability and fracture toughness (Wettstein et al., 2008).

Zirconia has the ability to transform from tetragonal to monoclinic crystal structure, resulting in a slight increase in crystal volume and creating favorable compressive stresses to resist crack propagation in a mechanism known as transformation toughening (Lughi and Sergo, 2010). It is well-established in literature that repeated loading in the patient's mouth can cause fracture of dental ceramics and affect the long-term reliability and stability of crowns. Therefore, it is better to test the fracture of the dental crowns under static and after being subjected to cyclic loading (Clausen et al., 2010). The reduction in the fracture strength of some dental ceramics after cyclic loading is caused by crack propagation (Erođlu and Gurbulak, 2013; Zhang and Lawn, 2004). Thus, the mechanical properties of the newly introduced 3D printed zirconia restorations are very important to withstand the mastication forces and their long-term survival. However, the data available for their properties and long-term functions is limited and needs more investigation (Elshiyab et al., 2017).

Accordingly, the aim of our study was to investigate the marginal gap, the internal fit and fracture resistance of 3D printed zirconia crowns and the effect of cyclic loading on their fracture resistance, compared to milled zirconia crowns. The first null hypothesis was that there would be no significant difference in the marginal adaptation between the 3D printed zirconia crowns compared to the milled crowns. The second null hypothesis postulated that the measurements of internal fit would be similar for both techniques used in this study. The third null hypothesis was that there would be no significant difference in the fracture resistance between the crowns fabricated by milling or 3D printed techniques. The fourth null hypothesis postulated that the cyclic loading would not affect the fracture resistance of zirconia crowns fabricated by both techniques.

1.2 Materials and Methods

A prepared upper premolar typodont tooth with a 1 mm deep chamfer finish line, 6 ° axial wall convergence and 1.5 mm occlusal reduction was used as an abutment (Fouda et al., 2023; Skjold et al., 2019). 30 epoxy stumps (Kemapoxy 150 3D, CMB, Wadi El Natroun, Egypt) were fabricated and allowed to set after taking impression replicas of the prepared tooth using addition silicone impression material putty soft and light consistency (Elite HD+, Zhermack, Rovigo, Italy) (Yucel et al., 2012). The epoxy stump was then scanned using an extraoral scanner (Medit T500, Medit, Seoul, Korea) and Exocad software (version 3.0, Darmstadt, Germany) was used to design the crowns.

In each group, 15 crowns were fabricated from 3 mol % yttria-stabilized zirconia. In the milled group (group M), IPS e.max ZirCAD LT (Ivoclar Vivadent, New York, USA) zirconia crowns were milled using a milling machine (DGSHAPE DWX-520 milling machine, Roland company, Willich, Germany) and subsequently sintered at up to 1530 °C in a zirconia furnace (Tabeo, MIHM VOGT, Stutensee Blankenloch, Germany). While the 3D printed zirconia crowns (group P) were fabricated using Lithoz 210 3Y (Lithoz GmbH, Vienna, Austria), then they were heat-treated in 3 successive furnaces (Nabertherm oven, Nabertherm GmbH, Lilienthal, Germany).

The crowns were checked for proper seating on the corresponding epoxy stumps using a sharp dental explorer. Subsequently, the fitting surface of all specimens was sand-blasted using 50 µm alumina particles at 2 bar for 10 seconds at a distance of 10 mm (Yim et al., 2023). The vertical marginal gap technique (VMGT) was used to evaluate the marginal gap (sample size N=10) using a stereomicroscope (Wild Leica M8, Leica Mikrosysteme, Heerbrugg, Switzerland) with a digital camera (Leica DFC 420 C, Leica Mikrosysteme, Wetzlar, Germany) at 50X magnification, the gap was measured according to the criteria proposed by Holmes et al. (1989) using Leica software (Leica LAS AF LITE 4.10.0). For standardization, 15 measurements were taken on each axial wall out of a total of 60 measurements and averaged for each sample (Faul et al., 2007).

To measure the internal fit of the crowns, the silicone replica technique (SRT) was used in which a low-viscosity silicone impression material (Honigum light, DMG, Hamburg, Germany) was injected into the fitting surface of the crown and the crown was seated

over the stump until fully set according to manufacturer's instruction. The crown was then removed while the light silicone body remained attached to the stump. To facilitate the removal of the light silicone body, a putty silicone material (Honigum putty soft, DMG, Hamburg, Germany) was applied over the light silicone body. It was then removed and cut into four parts buccolingually and mesiodistally.

Using a stereomicroscope (Wild Lecia M8) coupled with a Leica DFC 420 C digital camera (Leica Mikrosysteme) at 50X magnification, the internal fit and marginal gap were assessed by measuring the thickness of the light body at 16 different points using Leica software (Leica LAS AF LITE 4.10.0).

Then the crowns were cemented using a universal dual-cured resin luting cement (DUO-DUO-LINK, BISCO, Schaumburg, USA) and the epoxy stumps were fixed in copper tubes with auto-polymerized resin (Technovit 4004, Kulzer GmbH, Hanau, Germany).

Samples from each group (n=9) were subjected to static fracture load in a universal testing machine (Zwick Zmart-Pro, ZwickRoell GmbH & Co. KG, Ulm, Germany), with a metal rod with a spherical tip of 5 mm diameter applied directly perpendicular to the occlusal surface in the center of the crown at a speed of 1 mm/min as shown in Fig. 1. The other samples (N=6 for each group) were subjected to 1.2 million cycles (equivalent to 5 years of clinical service) using a commercial pneumatic setup (Dyna-Mess TP 5kN HF, DYNA-MESS Prüfsysteme GmbH, Stolberg, Germany) with a load between 20 N and 200 N at 2 Hz in distilled water 37 ± 2 °C (Elshiyab et al.;2017, Farga et al.;2016, Katzenbach et al.; 2021). The samples were fixed in a holder and placed in a small basin connected to a temperature-controlled reservoir of distilled water. Forces were applied perpendicular to the occlusal surface using a metal rod with a spherical tip of 5 mm diameter as shown in Fig. 2. The tempered distilled water was constantly pumped from the reservoir to the basin and overflowing fluid flows back through a second flexible tube.

The aged samples were examined for any cracks or fractures using a stereomicroscope (Wild Leica M8) equipped with a digital camera (Leica DFC 420 C) at 25X magnification. All the samples were free from any defects and were then loaded to fracture using the previously mentioned method.



Fig. 1: Static load applied until fracture using the universal testing machine. The Load applied perpendicular to the occlusal surface. Modified after Refaie et al.;2023.



Fig. 2: The basin filled with distilled water at a temperature of 37 ± 2 °C. Modified after Refaie et al.;2023.

Representative fractured samples were cleaned with alcohol in an ultrasonic bath and coated with a thin gold/platinum layer for 60 seconds using a sputter coater (Scancoat six, Edwards High Vacuum, England, UK). After sputtering, the samples were ready for fractographic analysis under a scanning electron microscope (SEM, Philips XL 30 CP, Philips, Eindhoven, The Netherlands) at an operating voltage of 10 kV and a spot size of 5 using secondary emission modes.

Numerical data were represented as mean with a 95 % confidence interval, standard deviation (SD), minimum and maximum values. Shapiro-Wilk's test was used to test for normality. The homogeneity of variances was tested using Levene's test. Data showed parametric distribution and variance homogeneity and were thus analyzed using a two-way ANOVA test. The significance level was set at $p < 0.05$ within all tests. Statistical analysis was performed with R statistical analysis software (R: A language and environment for statistical computing. R Foundation for Statistical Computing, Vienna, Austria. URL [https://www.R-project.org/...](https://www.R-project.org/)) version 4.1.3 for Windows (R Core Team, 2022).

1.3 Results

The results of intergroup comparisons for the marginal gap showed that the printed group had significantly higher mean values (80 μm) compared to the milled group (60 μm) with $p < 0.05$.

The results of intergroup comparisons of internal fit showed that the printed crowns had significantly higher mean values for MG, CG and OCC measurements (100 μm , 70 μm , and 300 μm respectively) than the milled crowns (60 μm , 50 μm and 210 μm respectively) with ($p < 0.001$), while the differences for AG were not statistically significant ($p > 0.05$).

The results of reliability and correlation analyses showed that there was a strong agreement ($\text{ICC} = 0.715$) and positive correlation ($r_s = 0.774$) between VMGT and SRT when measuring the marginal gap ($p < 0.001$).

The fracture resistance of the milled group was 1890 ± 191 N and 1642 ± 127 N before and after cyclic loading while that of the printed group was 1658 ± 333 N and 1224 ± 263 N respectively.

The results of two-way ANOVA for the effect of tested variables on fracture resistance showed that the fabrication technique ($p=0.002$) and the mechanical cycling loading ($p=0.001$) had a significant effect on fracture resistance while there was no significant interaction between tested variables ($p=0.325$).

SEM of the selected fractured samples showed similar fracture patterns where the cracks originated away from the point of load application and the direction of crack propagation started from the internal surface of the crown (the tensile zone) towards the external surface of the crown. It also showed the presence of twist hackles and arrest lines as shown in Fig.3 and 4.

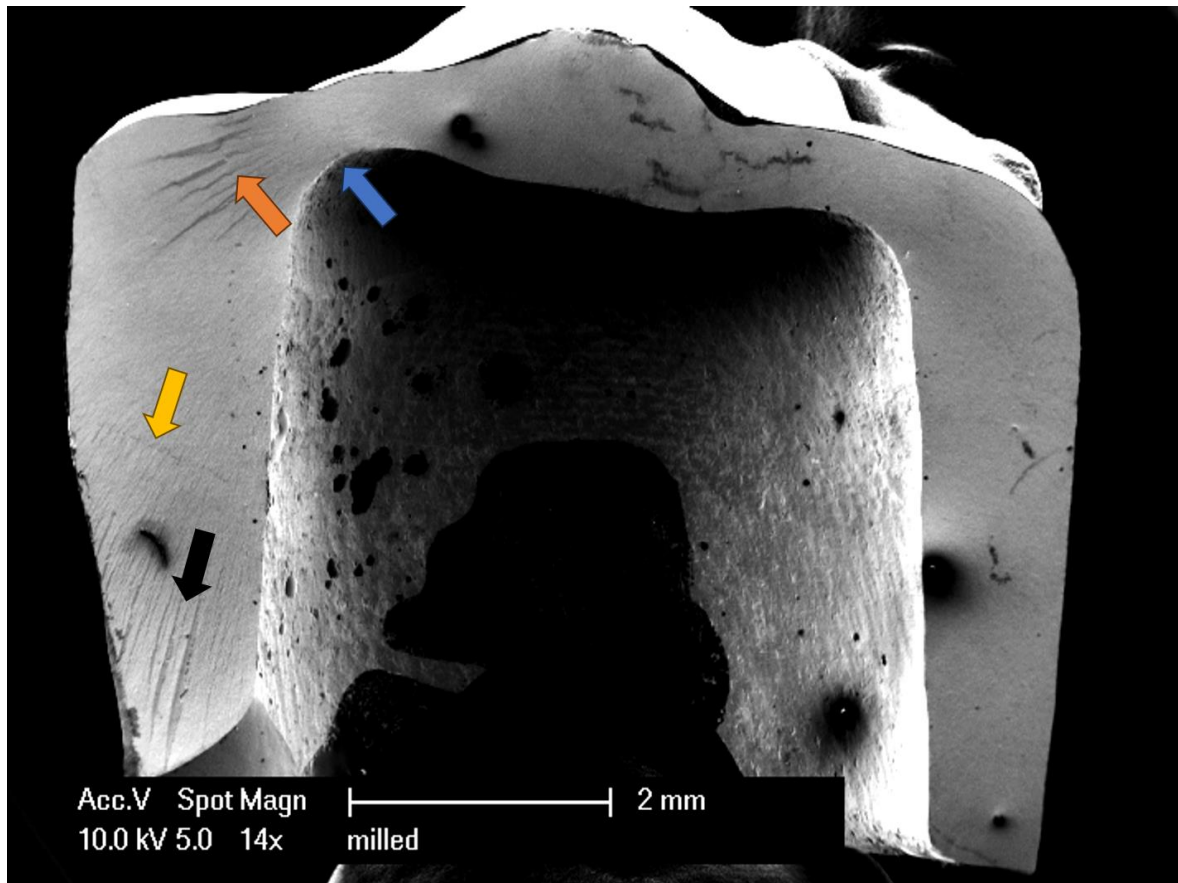


Fig. 3: A fractographic map of milled zirconia after fracture. Blue arrows show the crack's origin. Orange arrows show twist hackles. Yellow arrows show arrest lines. Black lines show the direction of crack propagation from the inner surface of the crowns and move outwards.

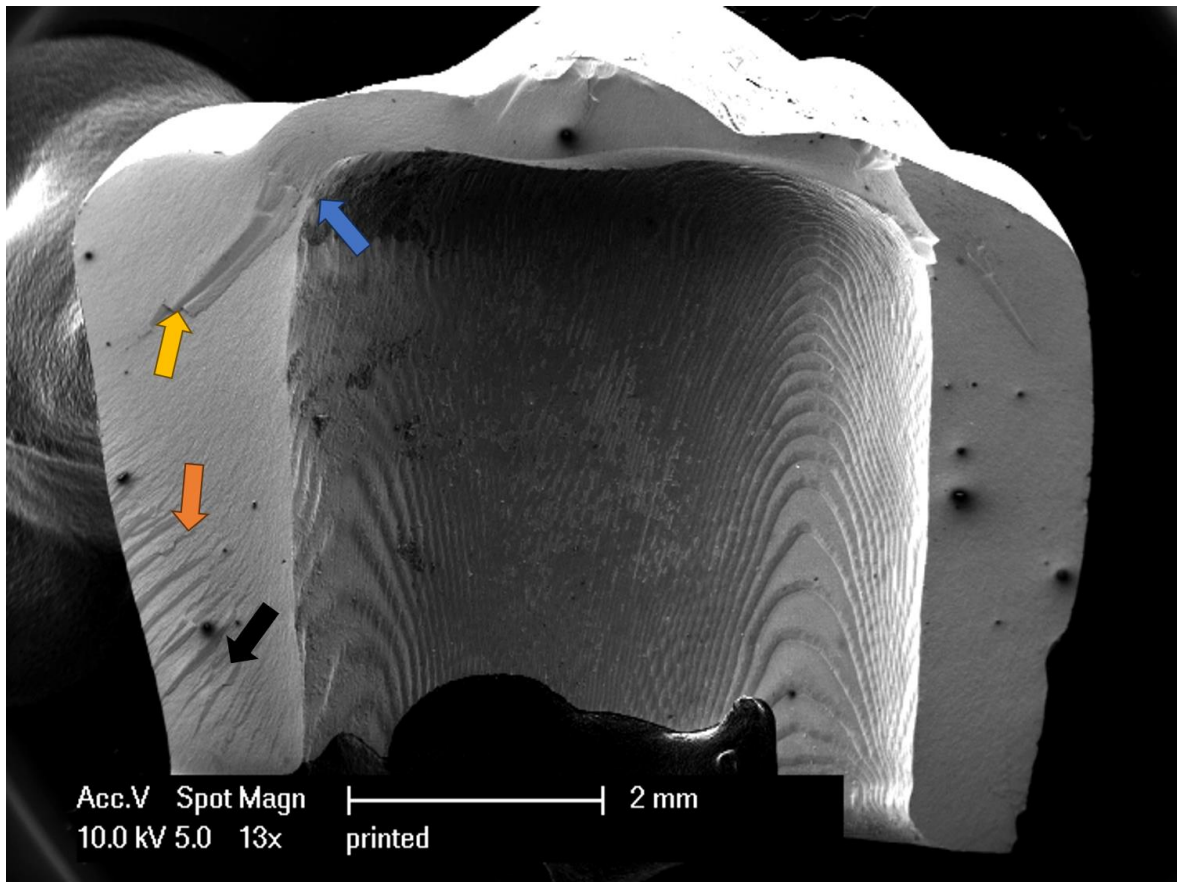


Fig.4: A fractographic map of printed zirconia after fracture. Blue arrows show the crack's origin. Orange arrows show twist hackles. Yellow arrows show arrest lines. Black lines show the direction of crack propagation from the inner surface of the crowns and move outwards.

1.4 Discussion

The first null hypothesis of the present study was rejected because the marginal gap of the milled crowns was lower than that of the 3D printed crowns. The second null hypothesis was rejected, as the milled crowns showed lower measurements of the MG, CG, and OG than the 3D printed crowns, except for the AG. The third null hypothesis was rejected as the milled crowns showed significantly higher fracture resistance than the 3D printed crowns. Also, the fourth null hypothesis was rejected, as the fracture resistance of zirconia crowns fabricated by both techniques was decreased after cyclic loading.

The presence of a marginal gap between the crown margin and the finish line leads to leakage and recurrent caries. The acceptable marginal gap should be below 120 μm , and the occlusal gap between 250 and 300 μm (Akbar et al., 2006; Euán et al., 2012).

All tested crowns fabricated with both techniques showed results below 110 μm , which is in the clinically acceptable range. The results of the marginal gap of the milled crowns (60 ± 20 μm) are in accordance with the results of many studies evaluating the marginal gap of monolithic zirconia crowns (24 to 110 μm) (Boitelle et al., 2014; Euán et al., 2014; Falahchai et al., 2021; Ji et al., 2015; Karataşlı et al., 2011). In addition, the result for the marginal gaps of the 3D printed crowns (80 ± 30 μm) agreed with results of a study by Ryu et al. (2020), who evaluated the marginal gap and internal fit of 3D printed resin crowns fabricated using different build directions.

A comparison between milled and printed crowns of the SRT results showed for MG, CG and OG that the milled crowns had lower values than the 3D printed crowns. The values of the marginal gap of the 3D printed crowns were higher using the SRT than that of the VMGT measurements, this could be due to the already present friction when the 3D printed crowns were seated on the epoxy stumps so when the light silicone impression material injected on the fitting surface caused an increase in MG, CG and OG. This can be overcome by increasing the cement gap by 10-15 μm compared to the cement gap used (70 μm), as suggested by (Ha and Cho, 2016). Increasing the cement gap of the 3D printed crowns during the design of the restoration can improve the fit of the restoration as indicated by (Baig et al., 2022; Ryu et al., 2020).

Regarding the fracture resistance of the tested crowns, all samples fractured at a value higher than the maximum biting force (450-520) N, which increases to 790 N in bruxism (Nishigawa et al., 2001; Özcan and Jonasch, 2018). All the samples showed no cracks or defects, which is consistent with the results stated by Rosentritt et al. (2020) and Spitznagel et al. (2021). Cyclic loading reduced the fracture resistance of the crowns fabricated by both groups (1658 N for the milled crowns and 1224 N for the 3D printed crowns) but was higher than the maximum biting forces. Thus, they could withstand up to 5 years of clinical service in the oral cavity.

The results of milled zirconia crowns showed higher fracture than those of 3D printed zirconia crowns, which is in agreement with the results of a study by (Zhai et al., 2023). This could be due to the presence of a higher amount of porosity in the 3D printed zirconia crowns caused by the entrapment of air bubbles in the paste of the 3D printed zirconia but without interlayer delamination, which affects the mechanical properties as stated by Branco et al., (2020).

The examined samples showed a similar fractographic pattern in the SEM where the fracture started away from the applied load in the inner surface of the crown (tensile zone) and propagated outwards. This could be related to the transverse expansion of the abutment material when the crown was subjected to occlusal loading due to the Poisson effect. The bulging resulted in the fracture of the crown (Schriwer et al., 2021). Epoxy resin and dentin have the same Poisson ratio, so the expected bulging of the epoxy is similar to the dentin during function when subjected to the same occlusal load (Yucel et al., 2012). The SEM also revealed small hackle lines merging to form twist hackles, as well as arrest lines.

This study has several limitations. First, the marginal gap and the internal fit of the 3D printed crowns must be measured between the different fabrication steps to allow for fine-tuning of the manufacturing process. Second, the use of resin abutment and the applied load was vertical without chewing cycles.

1.5 Summary

Objectives: The aim of this study was to evaluate the marginal gap of crowns fabricated by two different methods, the internal fit and the fracture resistance before and after cyclic mechanical loading of 3D printed zirconia crowns compared to milled zirconia crowns.

Materials and methods: Monolithic zirconia crowns (N=30) were manufactured using subtractive milling (group M) and 3D additive printing (group P). The marginal gap of 20 crowns was evaluated using the vertical marginal gap technique (VMGT). On the other hand, the silicone replica technique (SRT) was used to measure the internal fit at 16 reference points. Concerning the fracture resistance test, 9 crowns of each group were

fractured under static loading while the other 6 samples were subjected to cyclic loading for 1.2 million cycles before being subjected to static loading until fracture. Scanning electron microscope (SEM) fractographic analysis was carried out on fractured fragments of representative samples.

Results: Using VMGT, group P ($80\pm 30\ \mu\text{m}$) showed a higher mean marginal gap compared to group M ($60\pm 20\ \mu\text{m}$). The marginal gap measured with SRT showed that group P ($100\pm 10\ \mu\text{m}$) had significantly higher values compared to group M ($60\pm 10\ \mu\text{m}$). The internal fit showed a significant difference between the tested groups except for axial gap. The mean for fracture resistance of group M was 1890 N without cyclic loading and 1642 N after being subjected to cyclic loading and they were significantly higher than that of group P (1658 N and 1224 N respectively).

Conclusions: Although milled zirconia crowns showed better results, the 3D printed zirconia crowns showed clinically acceptable results in terms of marginal adaptation, internal fit and fracture resistance.

1.6 References for the English summary

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2. Publication

2.1 Marginal gap and internal fit of 3D printed versus milled monolithic zirconia crowns

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RESEARCH

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Marginal gap and internal fit of 3D printed versus milled monolithic zirconia crowns

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Abstract

Background This study aimed to evaluate and compare the marginal gap using two different methods and the internal fit of 3D printed and zirconia crowns.

Methods 3Y-TZP zirconia crowns ($n=20$) were manufactured using subtractive milling (group M) and 3D printed (group P). The marginal gap was measured at 60 points using vertical marginal gap technique (VMGT). On the other hand, the silicone replica technique (SRT) was used to evaluate the internal fit and was divided into 4 groups: marginal gap, cervical gap, axial gap, and occlusal gap where the thickness of light impression was measured at 16 references. The numerical data was tested for normality using Shapiro–Wilk’s test. They were found to be normally distributed and were analyzed using an independent t-test.

Results Using VMGT, group P had significantly higher mean marginal gap values of $80 \pm 30 \mu\text{m}$ compared to group M = $60 \pm 20 \mu\text{m}$ ($p < 0.001$). Also, with the SRT, the marginal gap of group P ($100 \pm 10 \mu\text{m}$) had significantly higher values compared to group M ($60 \pm 10 \mu\text{m}$). The internal fit showed significant difference between the tested groups except for Axial Gap.

Conclusions Although milled crowns showed better results. The 3D printed zirconia crowns offer clinically acceptable results in terms of marginal adaptation and internal fit. Both VMGT and SRT are reliable methods for the assessment of the marginal gap.

Keywords Yttria-stabilized zirconia crowns, 3D printing, Subtractive manufacturing, Marginal gap, Internal fit

Background

Monolithic zirconia restorations are among the most commonly used treatment options in modern dentistry owing to their biocompatibility, strength, and esthetics [1]. The conventional technique for the fabricating zirconia crowns is subtractive milling through computer-aided

design and computer-assisted manufacturing (CAD/CAM). Although subtractive milling technique is a very reliable method, it has many drawbacks such as the high production costs, the wear of the milling burs, the amount of wasted material, and the difficulty of milling complex geometries [2]. To overcome these drawbacks, many attempts have been made to manufacture dental crowns using additive manufacturing technologies (AM), so-called 3D printing techniques. AM techniques include selective laser sintering (SLS), selective laser melting (SLM), stereo-lithography (SLA), ink-jet printing (IJP), fused deposition modeling (FDM), and others [3].

Lithography-based Ceramics Manufacturing technique (LCM) could be used for the fabrication of ceramics. The CAD file of the desired design is virtually divided into very thin layers, the slurries

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(photosensitive ceramic suspensions) are deposited and cured using digital light processing (DLP), where the projected light source is used to cure the entire layer at once and the polymer network serves as a binder between the ceramic. The produced green bodies are a composite of polymerized binder with dispersed ceramic particles within, which is then cleaned with a direct stream of compressed air and suitable cleaning solvents to remove any excess or uncured raw material (slurry). Finally, the binder was removed and sintered to give the component its final properties [4].

The long-term prognosis of dental crowns depends on many aspects, with marginal adaptation being one of the most important factors that may lead to clinical failure [5, 6]. A marginal gap is defined as the vertical distance between the finish line of the preparation and the cervical margin of the restoration [7, 8]. This could be measured under high magnification with a stereomicroscope to measure the marginal gap from the crown margin to the finish line [9]. Poor marginal adaptation can lead to plaque accumulation, microleakage, recurrent caries, and periodontal disease [10]. A marginal gap between 50 and 120 μm is considered clinically acceptable [5]. Moreover, the internal fit is an important factor for the success of dental crowns, as deterioration of the internal fit of the crown will lead to a decrease in retention, a lack of rotational stability, and a reduction in fracture toughness [11].

There are also various techniques that can be used to measure the internal fit of dental crowns; such as repeated 3D scanning, which is a non-destructive method but requires special care to avoid errors [12, 13]. The cross-sectional method for cemented restoration is a well-known but destructive method [14, 15]. The triple scan protocol is also a non-destructive technique in which the fitting surface of the crown, the abutment and the crown seated on the abutment are scanned and overlapped using a software that allows measurement of the internal fit [16]. SRT is also a well-known technique for measuring the internal fit and the marginal gap [17–19].

Limited evidence is available on the marginal gap and the internal fit of 3D printed zirconia crowns as it is still a new technology. Therefore, it was the aim of this in vitro study to compare the aforementioned properties of 3D printed zirconia crowns with that of the commonly used CAD/CAM milling technique. The first null hypothesis was that there would be no significant difference in the marginal adaptation between the crowns fabricated by milling or 3D printing techniques. The second null hypothesis postulated that the measurements of internal fit would be similar for both techniques used in this study.

Methods

A typodont upper premolar tooth was prepared to create a 1 mm rounded deep chamfer with 6° convergence of the axial wall and 1.5 mm occlusal reduction. Addition silicone impression material (elite HD+, Zhermack SPA, Rovigo, Italy) putty soft and light consistency was used to make an impression replica. After the complete setting of the index, an epoxy die (KEMA-POXY 150 3D, CMB, Wadi El Natroun, Egypt) having the same modulus of elasticity as the human dentin was poured and allowed to set until full hardening. Then the epoxy die was scanned with an extra-oral scanner (Medit T500, MEDIT Corp., Seoul, Korea), and the produced STL files were used to digitally design the crowns using CAD software (exocad version 3.0, exocad GmbH, Darmstadt, Germany) with a 70 μm cement gap starting 1 mm from the finish line margin [20].

A power analysis was designed to obtain sufficient power for a two-sided statistical test of the null hypothesis that there is no difference between the tested groups. Assuming an alpha (α) level of 0.05 (5%), a beta (β) level of 0.2 (20%) (i.e., power = 80%), and an effect size ($d = 1.53$) calculated based on the results of a previous study [20], the predicted sample size (n) was found to be 16 samples i.e., 8 samples per group. Sample size calculation was performed using G*Power version 3.1.9.4 [21]. Thus, 20 zirconia crowns were fabricated using 3 mol-% yttria-stabilized zirconia using 2 different fabrication techniques with 10 crowns in each group.

In group M, IPS e.max ZirCAD LT (Ivoclar Vivadent, New York, USA) crowns were milled using a milling machine (DGSHAPE DWX-520 milling machine, Roland company, Willich, Germany) followed by sintering in a zirconia furnace (Tabeo, MIHM VOGT, Stutensee Blankenloch, Germany) up to 1530 °C. In group P, Lithoz 210 3Y (Lithoz GmbH, Vienna, Austria) crowns were 3D printed using a CeraFab7500 printer (Lithoz GmbH, Vienna, Austria), several small supports placed perpendicular to the lingual surface to hold and stabilize the crowns on the platform until the printing was complete. Then the printed crowns were heated in 3 consecutive furnaces (Nabertherm oven, Nabertherm GmbH, Lilienthal, Germany) as follows: 1) Preconditioning up to 120 °C for 134 h, 2) transfer to the second oven for debinding at up to 1000 °C for 103 h, and 3) sintering at up to 1450 °C for 17 h.

The internal surface of all crowns was not adjusted after sintering. The crowns were sandblasted using alumina particles of 50 μm at 2 bar for 10 s. The crowns were seated on the same epoxy die and a sharp dental explorer was used to check for proper seating.

Marginal gap evaluation (vertical marginal gap technique VMGT)

A stereomicroscope (Wild Lecia M8, Leica Mikrosysteme, Heerbrugg, Switzerland) with a digital camera (Leica DFC 420 C, Leica Mikrosysteme, Wetzlar, Germany) at 50X magnification was used to observe the marginal gap, which was measured using Leica software (Leica LAS AF LITE 4.10.0) as shown in Fig. 1. Calibration protocols of microscope were followed strictly before starting the measurements. The marginal gap was determined using the criteria proposed by Holmes et al. [7] who defined the vertical marginal gap as the distance between the crown margin to the edge of the finish line preparation. For standardization, each axial wall was divided into 3 equal parts, at each part 5 measurements for the marginal gaps were recorded, thus, 15 measurements for each axial wall were obtained, which were averaged to yield a single measurement for each sample [22].

Internal fit evaluation and marginal gap (Silicone replica technique SRT)

The internal fit was measured using the silicone replica technique in which a low viscosity silicone impression material (Honigum light, DMG, Hamburg, Germany) was injected into the fitting surface of the crown. The crown

was then seated over the abutment and pressed for 3 min and 30 s under a 5 kg load until the impression material was fully set according to the manufacturer's instructions. Subsequently, the crown was removed leaving the light silicone impression on the abutment representing the thickness of the cement space. A putty silicone material (Honigum putty soft, DMG, Hamburg, Germany) was then applied over the remaining light impression on the abutment to overcome the difficulties in handling and cutting the thin thickness of the light body. After setting, the silicone replica was removed and cut into four parts buccopalatally and mesiodistally using surgical blade no. 15 as shown in Fig. 2.

The thickness of the light body was observed using a stereomicroscope (Wild Lecia M8, Heerbrugg, Switzerland) coupled with a Leica DFC 420 C digital camera (Leica Mikrosysteme, Wetzlar, Germany) at 50X magnification to evaluate the internal fit and the marginal gap. The thickness of the light body silicone was measured at 16 different points, which were divided into 4 groups: Marginal Gap (MG), Cervical Gap (CG), Axial Gap (AG), and Occlusal Gap (OG). The internal fit was evaluated by the CG, AG, and OG [19]. Each point was measured using Leica software (Leica LAS AF LITE 4.10.0) as shown in Figs. 3 and 4.

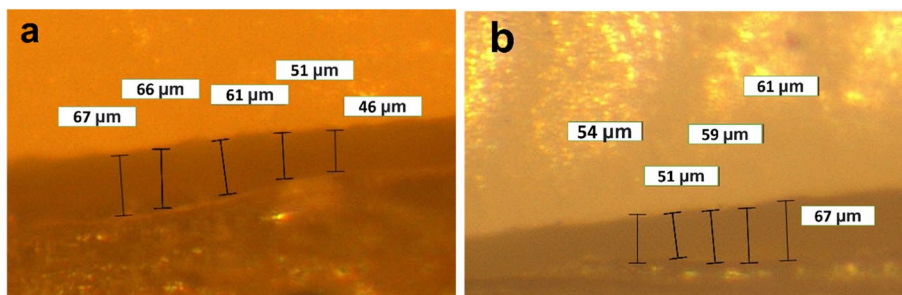


Fig. 1 Measurements of marginal gap. **a** Milled zirconia crown. **b** Printed zirconia crown

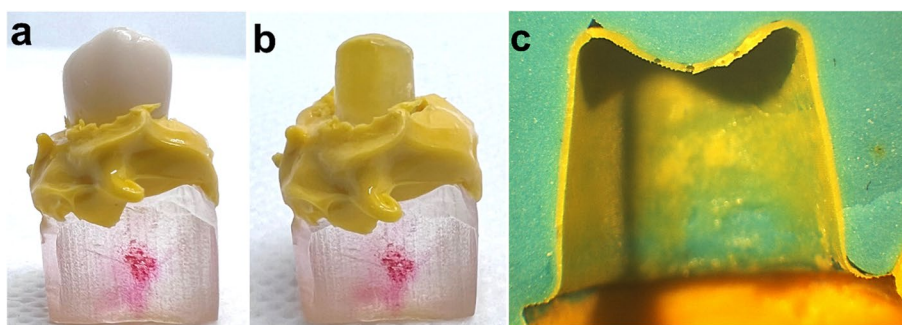


Fig. 2 Fabrication of silicone replica. **a** Low viscosity silicone impression material was injected in the fitting surface of the crown and then fitted to the epoxy die. **b** Crown was removed, and the low viscosity impression remains attached to the die. **c** After the putty was added, the replica was removed and cut buccolingually as in the figure, then it was cut mesiodistally

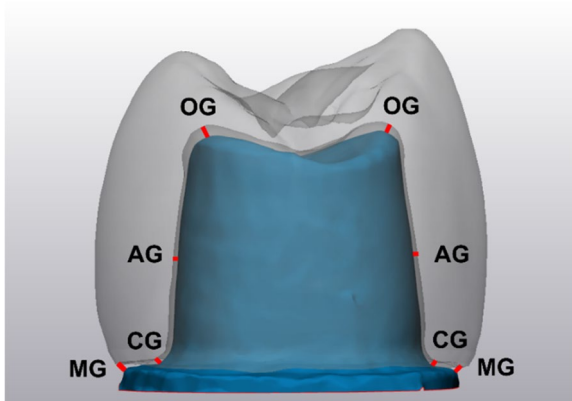


Fig. 3 Each section was measured at four different points: Marginal Gap (MG), Cervical Gap (CG), Axial Gap (AG), Occlusal Gap (OG)

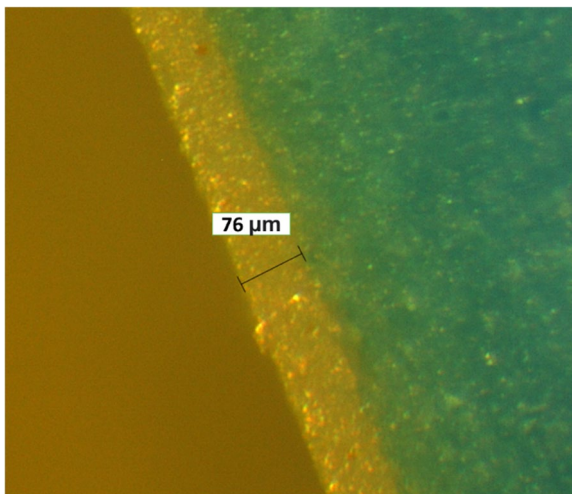


Fig. 4 Measuring the AG under 50 X magnification

Statistical analysis

Numerical data were tested for normality using Shapiro–Wilk’s test and represented as mean and standard deviation. Data were then analyzed using independent t-test, reliability analysis was performed with the Intra-class correlation coefficient (ICC). Correlation analysis was performed using Spearman’s rank order correlation coefficient (rs). The significance level was set at $p < 0.05$ for all tests. Statistical analysis was performed with R statistical analysis software (R Core Team 2023) version 4.1.3 for Windows.

Results

The results of intergroup comparisons for marginal gap are presented in Table 1. It was found that group P had significantly higher mean values of 80 μm compared to group M that showed a mean marginal gap of 60 μm ($p < 0.05$).

Table 1 Intergroup comparisons of marginal gap (in μm) of the 3D printed (P) and milled (M) groups

Point	Marginal gap in μm (Mean \pm SD)		t-value	p-value
	Group M	Group P		
Average	60 \pm 20	80 \pm 30	3.59	< 0.001*

* significant ($p < 0.05$)

The results of intergroup comparisons of internal fit showed that group P had significantly higher mean values than group M for MG, CG and OCC measurements ($p < 0.001$), while the differences for axial measurements were not statistically significant ($p > 0.05$). The Mean and standard deviations for internal fit are illustrated in Fig. 5.

The results of reliability and correlation analyses showed that there was a strong agreement (ICC = 0.715) and positive correlation ($r_s = 0.774$) between the marginal gap (VMGT) using stereomicroscope and the marginal gap using the silicone replica method (MG) ($p < 0.001$) as shown in Fig. 6.

Discussion

In this study, the first null hypothesis was rejected because the measurements of the marginal gap were lower in the milled group (M) than in the 3D printed group (P). The second null hypothesis was rejected, as the measurements of the marginal gap, cervical gap, and occlusal gap were lower in the milled crowns than in the 3D printed crowns, except for that the axial gap.

A large marginal gap between the restoration and the tooth leads to leakage and recurrent caries. Thus, the presence of a marginal gap is one of the most important aspects to be considered when choosing the method for fabricating a dental crown, especially when new technologies are used. Despite careful preparation, there is always a gap between the margin of a full coverage restoration and the finish line of the prepared tooth. In addition, the internal fit of restoration is important for the retention and resistance of the crown. The literature reports that the normal acceptable marginal gap should be below 120 μm and the occlusal gap between 250 and 300 μm [5, 23].

At the beginning of this research, micro-CT was used to measure the marginal gap and the internal fit, but then it was excluded due to some limitations that made the readings inaccurate. It was extremely difficult to define specific points at the margin of the restoration and the finish line, which could be due to the radiation absorption coefficient and artificial defects caused by the reflection of rays [21, 24]. Accordingly, two of the most common techniques were used to measure the marginal gap and the internal fit. In the first method, a

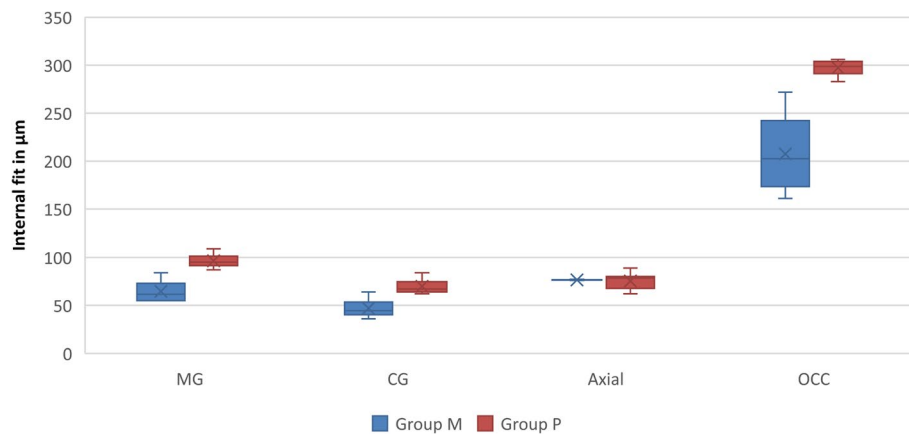


Fig. 5 Box plot showing values of internal fit measured at different points

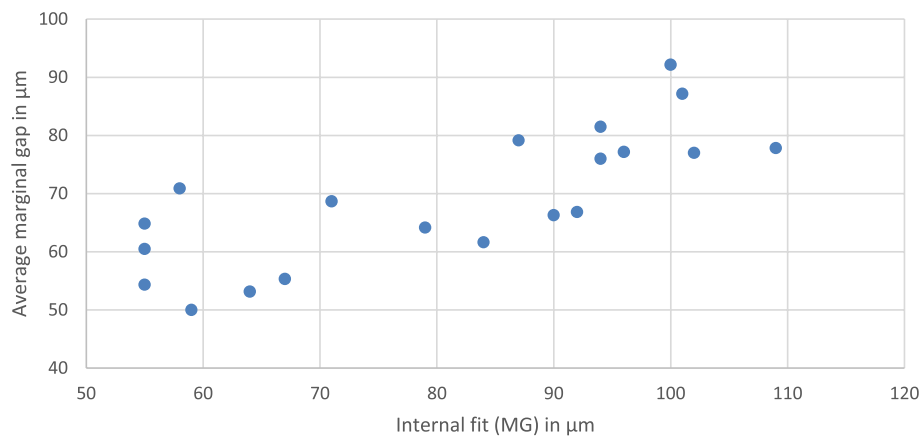


Fig. 6 Scatter plot showing the correlation between average marginal gap under stereomicroscope and MG using silicone replica technique

stereomicroscope was used to measure the marginal gap between the crown margin and the finish line [9]. In the second method, the silicone replica technique using light body silicone impression material was used to determine the cement space thickness in order to evaluate the internal fit as well as the marginal gap of the restoration. Many authors compared the use of SRT to other techniques, and they found that it offers reliable results with the advantage of being used in both in vivo and in vitro studies as it is a non-destructive method [21, 25, 26]. The results of this study showed strong agreement between the two methods used in measuring the marginal gap.

The values of the marginal gaps of all zirconia crowns manufactured with both techniques were below $110 \mu\text{m}$, thus all the specimens were within the clinically acceptable range. The range of the marginal gap of the milled crowns was $60 \pm 20 \mu\text{m}$. This is in agreement with the results of many authors who investigated the marginal gap of monolithic zirconia crowns, where the gap ranged from 24 to $110 \mu\text{m}$ for CAD/CAM restorations [9,

27–30]. Moreover, the marginal gaps of the 3D printed crowns were in the range of $80 \pm 30 \mu\text{m}$, which is in accordance with Ryu et al. [19] who compared the effect of different build directions on the marginal and internal fits of 3D printed resin crowns.

The mean marginal gap of the milled crowns in this study was significantly lower than that of the 3D printed zirconia crowns. This could be related to over-polymerization of the material during fabrication due to light scattering, which means that more material hardens than intended. However, the manufacturer set a contour offset of $40 \mu\text{m}$ in the printer software to counteract this effect. Therefore, additional studies are needed to investigate the effect of changing the contour offset value during the manufacturing of zirconia crowns on enhancing the marginal adaptation.

For the SRT, the results of the marginal gap, cervical, and occlusal gap between milled and printed crowns showed that the milled crowns had lower values than the 3D printed crowns. Comparing the marginal gap

results of the 3D printed crowns between the two measurement methods used, the values were higher in the SRT. This could be due to the preexisting friction noticed when the 3D printed crowns were seated on the epoxy die during the checking phase so when the light impression was injected into the fitting surface, it caused the increase in the marginal gap, cervical gap and the occlusal gap in 3D printed crowns. This problem can be solved by increasing the cement gap by more than 70 μm by 10–15 μm and testing the results as suggested by Ha et al. [21]. In many studies using the 3D printing technique in the fabrication of resin-based crowns, it was found that the measured internal fits were 3–4 times the cement gap used in designing. Thus, increasing the cement gap value during designing the restoration with the CAD software can improve the fit of the fabricated 3D printed crowns [19, 31].

This study has several limitations. First, the marginal gap and the internal fit of the 3D printed crowns were not measured between the different manufacturing steps. Future studies are recommended to allow fine-tuning of the manufacturing process. Second, the use of natural human teeth instead of resin abutment would simulate the clinical conditions, although resin abutment was used in this study to allow standardization.

Additional studies are needed to determine the optimal cement space value for the CAD design, which is required to reduce marginal gap without compromising the internal fit and to provide maximum retention and resistance for better clinical outcomes.

Conclusions

The 3D printed zirconia crowns showed higher values for marginal gap and internal fit compared to the milled zirconia crowns, but within the clinically acceptable range. The 3D printing technique showed promising results within the clinically acceptable range for marginal gap and internal fit. Thus, 3D printed monolithic zirconia crowns can be considered a clinically acceptable alternative in terms of marginal adaptation and the internal fit. The evaluation of marginal gap could be measured with both the VMGT and the SRT, with both techniques showing reliable and comparable results.

Abbreviations

VMGT	Vertical marginal gap technique
SRT	Silicone replica technique
CAD/CAM	Computer aided designing / Computer aid manufacturing
AM	Additive manufacturing
LCM	Lithography-based Ceramics Manufacturing technique
DLP	Digital light processing
MG	Marginal gap
CG	Cervical gap
AG	Axial gap
OCC	Occlusal gap

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Authors' contributions

Conceptualization: AR. Methodology, Investigation, Formal analysis: AR, AF, LS. Resources: CB, AR. Writing – original draft preparation: AR. Writing – review and editing: AF, CB, LS. Supervision: CB, LS.

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Availability of data and materials

The datasets supporting the conclusions of this article are included within the article.

Declarations

Ethics approval and consent to participate

Not applicable.

Consent for publication

Not applicable.

Competing interests

The authors declare no competing interests.

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2.2 The Effect of Cyclic Loading on the Fracture Resistance of 3D Printed and CAD/CAM Milled Zirconia Crowns - An In-Vitro Study

Refaie A, Bourauel C, Fouda A M, Keilig L, Singer L. **The Effect of Cyclic Loading on the Fracture Resistance of 3D Printed and CAD/CAM Milled Zirconia Crowns - An In-Vitro Study.** Clin Oral Investig. 2023.

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The effect of cyclic loading on the fracture resistance of 3D-printed and CAD/CAM milled zirconia crowns—an in vitro study

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Abstract

Objectives The aim of this study was to evaluate the effect of cyclic mechanical loading on the fracture resistance of 3D-printed zirconia crowns in comparison to milled zirconia crowns.

Materials and methods Monolithic zirconia crowns ($n=30$) were manufactured using subtractive milling (group M) and 3D additive printing (group P). Nine samples of each group were fractured under one-time loading while the other 6 samples were subjected to cyclic loading for 1.2 million cycles before being subjected to one-time loading until fracture. Scanning electron microscope (SEM) fractographic analysis was carried out on fractured fragments of representative samples.

Results The mean for fracture resistance of group M was 1890 N without cyclic loading and 1642 N after being subjected to cyclic loading, and they were significantly higher than that of group P (1658 N and 1224 N respectively).

Conclusions The fabrication technique and cyclic loading affect the fracture resistance of zirconia crowns. Although the fracture resistance values for the 3D-printed crowns were lower than those of the milled, still they are higher than the masticatory forces and thus could be considered being clinically acceptable.

Clinical relevance Concerning fracture resistance, 3D-printed crowns can withstand the masticatory forces for the long term without any cracks or failure.

Keywords 3D printing · Zirconia · Fracture resistance · Cyclic loading

Introduction

Patients and dentists have been looking for metal-free tooth-colored restorations due to the increased esthetic demands, the more conservative preparations, and the high concerns of

toxic and allergic reactions to certain alloys [1]. Yttria partially stabilized zirconia (Y-TZP) ceramics have been widely used because of their desirable esthetics, biocompatibility, superior fracture strength, and fracture toughness compared to other ceramics used in dentistry [2]. Core substructures can be fabricated, then veneered with glass ceramics in layering, press, or computer-aided design/computer-aided manufacturing (CAD/CAM) techniques. However, the veneer is directly exposed to chewing, clenching, and moisture, which might weaken it and result in cracking or chipping [3]. To overcome this problem, monolithic zirconia dental restorations with full anatomic contour have been developed without the need for adding a veneer layer [4].

The development of these materials and the CAD/CAM fabrication techniques has created a wide range of applications for dental restorations with conservative tooth preparation, such as partial coverage restorations, crowns, and bridges [5]. Monolithic zirconia dental restorations are usually fabricated by subtractive milling. This technique has some drawbacks such as the difficulty of milling a thin margin restoration without the possibility of chipping and

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the difficulty of fabricating a restoration with deep grooves and complex structures [6–8]. In addition, this technique produces a lot of material waste, deterioration of the burs, and high production costs [9].

The fast expansion and the development of materials and techniques used in additive manufacturing allowed the possibility of the production of monolithic zirconia dental restorations from a variety of materials; additive manufacturing techniques include selective laser sintering (SLS), selective laser melting (SLM), stereolithography (SLA), ink-jet printing (IJP), and fused deposition modeling (FDM) [10]. In this study, lithography-based ceramic manufacturing (LCM) technology was used for the fabrication of 3D-printed zirconia crowns because this is the only available and most advanced approach to fabricate a fully anatomical 3D-printed zirconia crown [11].

The fracture resistance of all-ceramic restorations is one of the major concerns in clinical applications of these materials, and it is influenced by many factors such as surface roughness, elastic modulus, crack resistance, and fabrication technique [12, 13]. Zirconia can resist crack propagation by transforming from tetragonal to monoclinic causing a slight increase in the volume of crystals and generating favorable compressive stresses around the crack in a mechanism called transformation toughening [14]. However, zirconia may undergo low-temperature degradation (LTD) in aqueous environment over time, which causes a reduction in its mechanical properties [15].

It has been well-established in the literature that fracture is the main cause of failure of dental ceramics after years of service inside the oral cavity, as they are subjected to repeated loads in the patient's mouth, which can influence the long-term reliability of the crowns. Consequently, to estimate the mechanical performance of zirconia crowns, it is not sufficient to test them under one-time loading only, cyclic loading must be tested as well [16]. Several studies have reported a reduction in flexural stress and toughness of some dental ceramics after cyclic loading [17]. The reduction of mechanical strength due to cyclic load is caused by the propagation of cracks at existing defects in the microstructure [1]. Thus, the mechanical properties of the newly introduced 3D-printed zirconia restorations are very important to withstand the mastication forces and their long-term survival. However, the data available for their properties and long-term functions is limited and needs more investigation [18].

Accordingly, the aim of our study was to evaluate the fracture resistance of 3D-printed zirconia crowns and the effect of cyclic loading in an aqueous solution on their fracture resistance, compared to milled zirconia crowns. The first null hypothesis was that there would be no significant difference in the fracture resistance between the crowns fabricated by milling or 3D-printed techniques. The second

null hypothesis postulated that the cyclic loading would not affect the fracture resistance of zirconia crowns fabricated by both techniques.

Materials and methods

Preparation criteria

An upper premolar typodont tooth was prepared using tapered stone with a round end to create a deep chamfer finish line with 1 mm thickness, axial wall convergence of 6%, and 1.5 mm occlusal reduction [19, 20].

Thirty impression replicas were taken for the prepared tooth using addition silicone impression material (Elite HD +, Zhermack, Rovigo, Italy) and putty soft and light consistency to pour 30 epoxy stumps (Kemapoxy 150 3D, CMB, Egypt) that were allowed to set until full hardening [21].

Scanning designing and fabrication of zirconia crowns

After scanning the epoxy stump with an extraoral scanner (Medit T500, Medit, Seoul, Korea), the crowns were digitally designed using exocad software (version 3.0, Darmstadt, Germany).

A total of 30 zirconia crowns (3 mol % yttria-stabilized zirconia) were fabricated using 2 different fabrication techniques with 15 crowns in each group. As a control group (group M), IPS e.max ZirCAD LT (Ivoclar Vivadent, New York, USA) zirconia crowns were milled using a milling machine (DGSHAPE DWX-520 milling machine, Roland Company, Willich, Germany); then, they were sintered in a zirconia furnace (Tabeo, MIHM VOGT, Stutensee Blankenloch, Germany) up to 1530 °C, while in group P, Lithoz 210 3Y (Lithoz GmbH, Vienna, Austria) zirconia crowns were 3D printed using a CeraFab7500 printer (Lithoz GmbH, Vienna, Austria); then, they were preconditioned up to 120 °C for 134 h, then debound at up to 1000 °C for 103 h, then sintered at up to 1450 °C for 17 h in 3 consecutive furnaces (Nabertherm oven, Nabertherm GmbH, Lilienthal, Germany). The materials used for each group in the study and the sintering parameters are listed in Tables 1 and 2.

A power analysis study was performed using the G power statistical power analysis program (version 3.1.9.4, University of Düsseldorf, Germany) to estimate the sample size. A total sample size of 30 samples (15 in each group, 9 to be used for measuring fracture without fatigue and 6 for measuring fracture after cyclic fatigue) was found to be sufficient to detect a large effect size (d) ranging from 1.46 to 1.82, with an actual power ($1 - \beta$ error) of 0.8 (80%) and

a significance level (α error) of 0.05 (5%) for a two-sided hypothesis test [22].

The study design and grouping are illustrated in Fig. 1.

Crown cementation

A sharp dental explorer was used to check for proper seating of the crowns on the corresponding epoxy dies before

Table 1 Materials used for each group in the study

Group name	Product name	Manufacturer	Composition
Milled group (group M)	IPS e.max Zir-CAD LT, Ivoclar Vivadent, USA LOT: Y43302	Ivoclar Vivadent AG, USA	Zirconium oxide (ZrO_2) 88.0–95.5 wt%, yttrium oxide (Y_2O_3) > 4.5– \leq 6.0 wt%, hafnium oxide (HfO_2) \leq 5.0 wt%, aluminum oxide (Al_2O_3) \leq 1.0 wt%, other oxides for coloring \leq 1.0 wt%
Printed group (group P)	Lithacon 3Y 210, Lithoz, Vienna, Austria LOT: AB0722038	Lithoz GmbH, Vienna, Austria	Zirconium oxide (ZrO_2) 3 mol-% Y_2O_3 stabilized

Table 2 Firing parameters of zirconia crowns

Group		Furnace	Highest temp in °C	Cycle time
M	Sintering	Tabeo, MIHM VOGT, Stutensee Blankenloch, Germany	1530	400 min (6 h and 40 min)
P	Preconditioning	Nabertherm TR 60, Nabertherm GmbH, Lilienthal, Germany	120	134 h
	Debinding	Nabertherm L 40/11 BO, Nabertherm GmbH, Lilienthal, Germany	1000	103 h
	Sintering	Nabertherm HT 40/17, Nabertherm GmbH, Lilienthal, Germany	1450	17 h

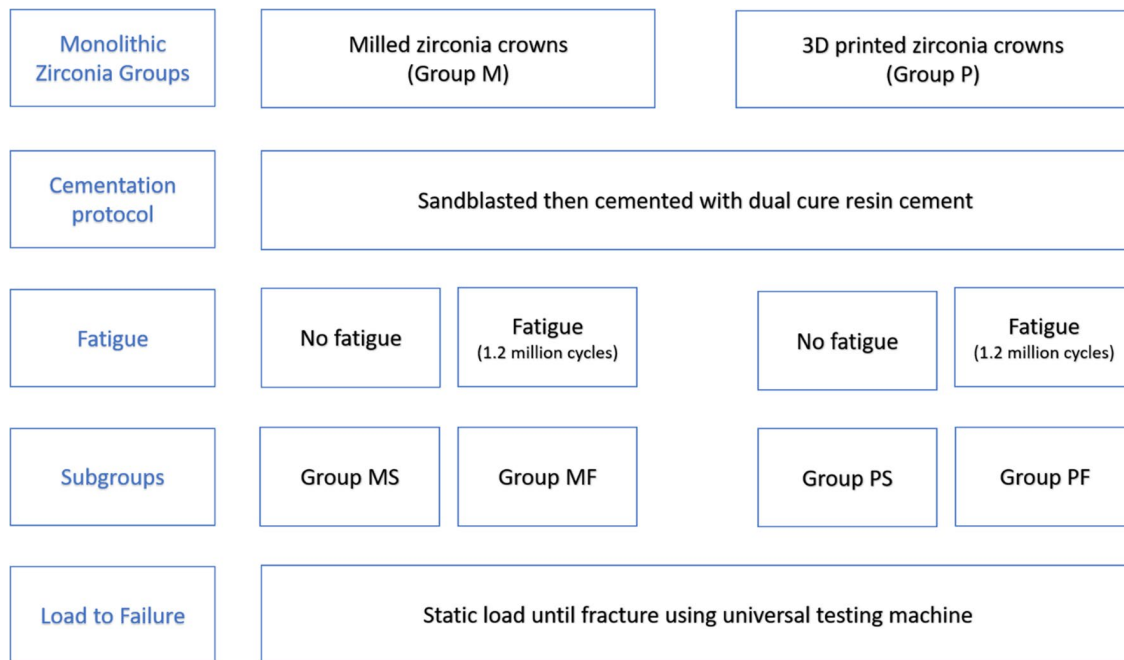


Fig. 1 Schematic diagram showing the study groups

cementation. The fitting surface of the crowns was sand-blasted using 50- μm alumina particles at 2 bar for 10 s at a distance of 10 mm [23], while no surface finish was made to the outer surface after fabrication. A universal dual-cured resin luting cement (Duo-Link, Bisco, Schaumburg, USA) was auto-mixed and injected into the crown fitting surface; then, the crowns were seated on the epoxy dies. Initial light curing was made for 3 s; then, the excess cement was removed using a sharp dental explorer followed by curing for 20 s on each surface.

One-time loading and cyclic loading fracture resistance testing

After the cementation of the dental crowns, the epoxy dies were trimmed to fit centrally in copper tubes and fixed with auto-polymerized resin (Technovit 4004, Kulzer GmbH, Hanau, Germany). Samples of each group ($n=9$, MS, PS) were loaded using a metal stylus with a 5-mm-diameter spherical tip with a speed of 1 mm/min which was applied directly to the center of each crown and perpendicular to the occlusal surface until fracture using a universal testing machine (Zwick Zmart-Pro, ZwickRoell GmbH & Co. KG, Ulm, Germany) (see Fig. 2).

The other 6 samples of each group (MF, PF) were subjected to dynamic mechanical loading using a commercial pneumatic setup ("Dyna-Mess TP 5kN HF", DYNA-MESS Prüfsysteme GmbH, Stolberg, Germany). 1.2 million cycles were applied with a load between 20 and 200 N at 2 Hz in distilled water $37 \pm 2^\circ\text{C}$ to simulate approximately 5 clinical years [18, 24, 25]. Specimens were fixed in a specimen holder positioned so that the forces could act directly on the center and perpendicular to the occlusal



Fig. 2 Load to fracture test using the universal testing machine. The piston with a tip diameter of 5 mm is applied perpendicular to the occlusal surface and driven at a crosshead speed of 1 mm/min

surface using a metal stylus with a 5-mm-diameter spherical tip in a small basin connected by a temperature-controlled reservoir of purified water as shown in Fig. 3. The tempered purified water was constantly pumped from the reservoir to the basin and overflowing fluid flows back through a second flexible tube. Specimens were examined using a stereomicroscope (Wild Leica M8, Heerbrugg, Switzerland) equipped with a digital camera (Leica DFC 420 C, Leica Mikrosysteme, Wetzlar, Germany) at $\times 25$ magnification. Samples with any cracks or defects were discarded. All specimens subjected to dynamic loading were free from any cracks, and then, they were loaded until fracture using the previously mentioned method.

Fractographic analysis

Representative fractured crown samples from each group were selected, cleaned using alcohol in an ultrasonic cleaner, and coated with a thin gold/platinum layer using a sputter coater (Scancoat Six, Edwards High Vacuum, England, UK) for 60 s. After sputtering, the samples were ready to be examined under a scanning electron microscope (SEM, Philips XL 30 CP, Philips, Eindhoven, Netherlands) at an operating voltage of 10 kV and a spot size of 5 using secondary emission modes.

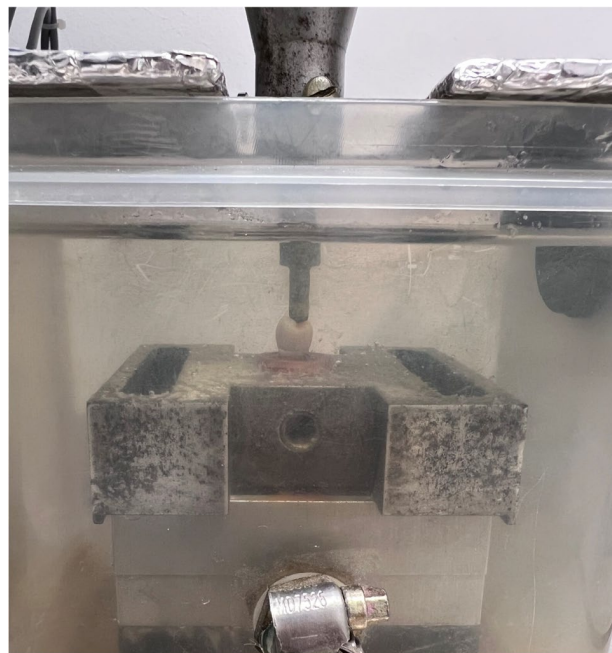


Fig. 3 The basin used to realize the wet environment with a temperature of $37 \pm 2^\circ\text{C}$

Statistical analysis

Numerical data were represented as mean with a 95% confidence interval, standard deviation (SD), and minimum and maximum values. Shapiro–Wilk’s test was used to test for normality. The homogeneity of variances was tested using Levene’s test. Data showed parametric distribution and variance homogeneity and were analyzed using a two-way ANOVA test. The significance level was set at $p < 0.05$ within all tests. Statistical analysis was performed with R statistical analysis software (R: A language and environment for statistical computing. R Foundation for Statistical Computing, Vienna, Austria. URL <https://www.R-project.org/>...) version 4.1.3 for Windows (R Core Team 2022).

Results

Descriptive statistics for fracture resistance values are presented in Fig. 4. It showed that the mean for the fracture resistance of group MS was 1890 ± 191 N and it decreased to 1642 ± 127 N after being subjected to dynamic mechanical loading in group MF while the fracture resistance of group PS was 1658 ± 333 N and decreased after dynamic mechanical loading to 1224 ± 263 N in group PF. Results of two-way ANOVA for the effect of tested variables on fracture resistance are presented in Table 3. It was found that the fabrication technique ($p = 0.002$) and the mechanical cycling loading ($p = 0.001$) had a significant effect on fracture resistance while there was no significant interaction between tested variables ($p = 0.325$).

Fig. 4 Box plot showing fracture resistance values in N

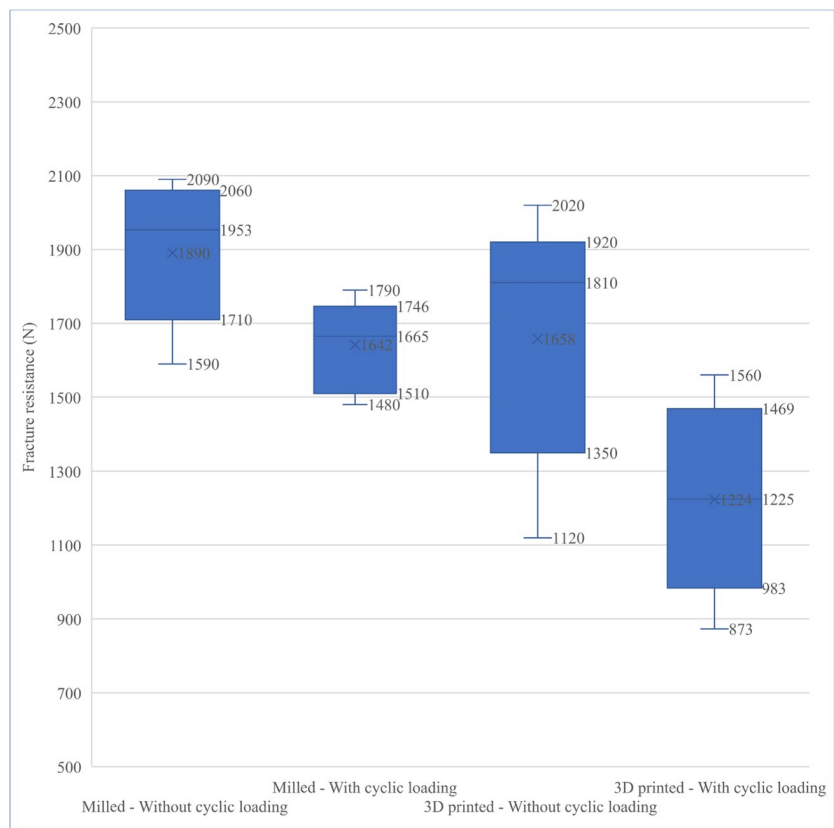


Table 3 Two-ANOVA for fracture resistance, values in bold indicate significant difference

Parameter	Sum of squares	df	Mean square	f-value	p-value
Fabrication technique	705,964	1	705,964	11.4	0.002*
Cyclic loading	838,914	1	838,914	13.6	0.001*
Technique * loading	62,163	1	62,163	1	0.325
Error	1,606,803	26	61,800		

*Significant ($p < 0.05$)

SEM showed similar fracture patterns in the selected samples, the cracks in all the groups originated away from the point of load application, and the direction of crack propagation started from the tensile zone at the internal surface and propagated outwards to the external surface of the crown. Twist hackles and arrest lines are present as shown in Figs. 5 and 6.

Discussion

The first null hypothesis of the present study that there would be no significant difference in the fracture resistance between the crowns fabricated by milling and 3D-printing techniques was rejected. Also, the second null hypothesis was rejected, as the cyclic loading decreased the fracture resistance of zirconia crowns fabricated by both techniques.

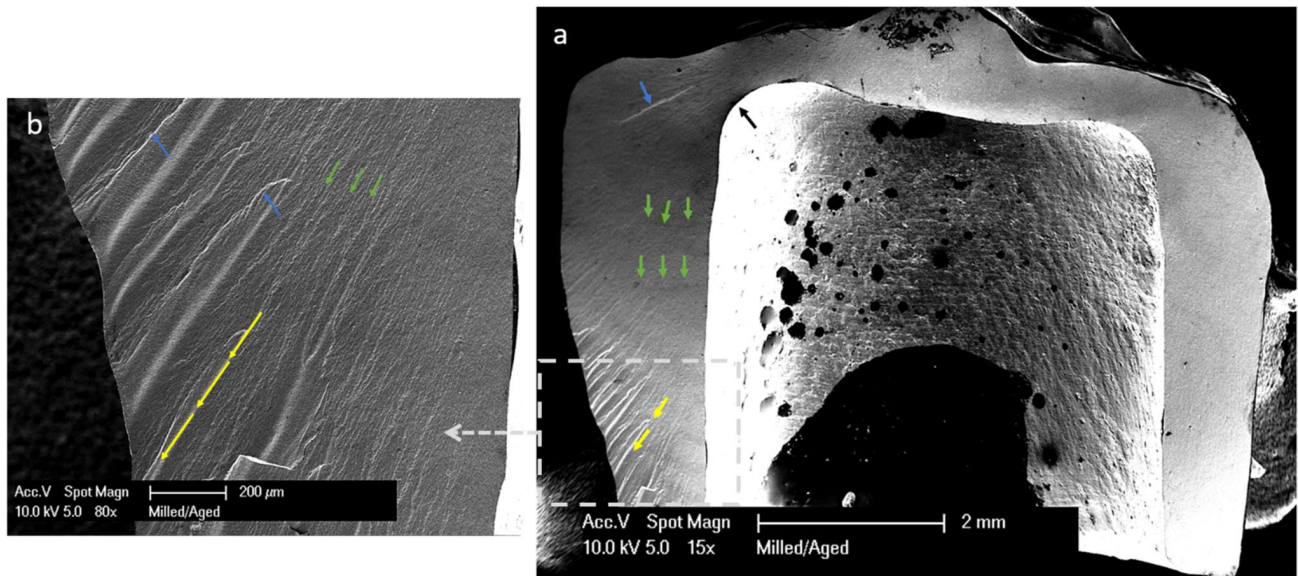


Fig. 5 A fractographic map of milled zirconia after dynamic loading. **a** SEM magnified at 15 \times . **b** Detailed image magnified at 80 \times . Black arrows show crack origin. Blue arrows show twist hackles. Green

arrows show arrest lines. Yellow lines show the direction of crack propagation from the inner surface of the crowns and move outwards

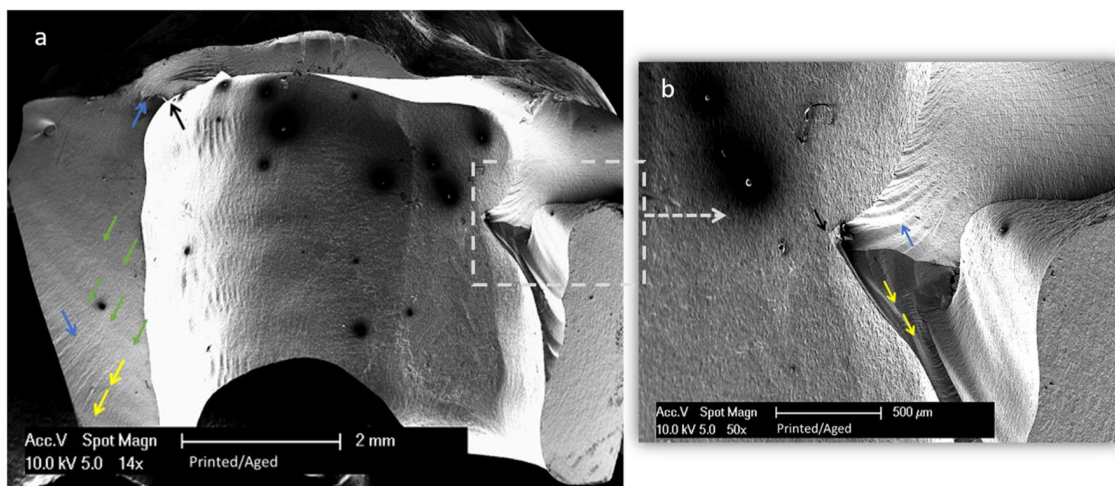


Fig. 6 A fractographic map of 3D-printed zirconia after dynamic loading. **a** SEM magnified at 14 \times . **b** Detailed image magnified at 50 \times . Black arrows show crack origin. Blue arrows show twist hackles. Green

arrows show arrest lines. Yellow lines show the direction of crack propagation from the inner surface of the crowns and move outwards

In the present study, premolars were used as they require an esthetic restoration that can withstand high masticatory forces [19, 20]. Similar stumps and crowns were used for standardization. Epoxy resin was used to prepare the stumps as it has an elastic modulus of 11.8 GPa, which is close to that of dentin (18.6 GPa) [21, 26]. In this regard, the elastic modulus of the stump has a significant effect on the fracture resistance of all-ceramic crowns [27], as it was reported that increasing the elastic modulus of the stump material leads to an increase in the fracture resistance of all-ceramic crowns [28]. Many studies have evaluated the fracture strength of all ceramic crowns using stainless steel [27], copper [29], acrylic resin [30], epoxy resin [31, 32], and dentin [33]. According to Yucel et al. [21], the stress distributions of dentin and epoxy resin stump could simulate the clinical conditions with realistic fracture strength values compared to those of stainless steel and copper.

Accelerated aging tests provide essential information on lifetime predictions of ceramic restorations; the applied fatigue protocol with 1.2 million cycles is equivalent to 5 years of clinical service for dental restorations [34]. The cyclic loading was done at a frequency of 2 Hz as the range of the chewing activity is between 0.94 and 2.17 Hz [35].

In this study, the immediate fracture resistance of all crowns (1890 ± 191 N for the milled crowns and 1658 ± 333 N for the 3D-printed crowns) exceeded 790 N, which is higher than the maximum biting forces (450–520 N) that increases to 790 N in bruxism [36, 37]. After cyclic loading, all the specimens showed no cracks or catastrophic failure which is similar to other studies that tested zirconia crowns [34, 38]. The fracture resistance of the crowns was reduced after cyclic loading (1658 N for the milled crowns and 1224 N for the 3D-printed crowns); however, it was higher than the maximum biting forces. Accordingly, crowns fabricated by both milling and 3D-printing techniques could withstand up to 5 years of clinical service in the oral cavity.

Milled zirconia crowns showed significantly higher fracture resistance than the 3D-printed zirconia crowns. These results were consistent with the results of Zhai et al. [39].

This may be contributed to the higher amount of porosity in the 3D-printed zirconia crowns. This effect has been widely studied by Branco et al. [40], who found no features of interlayer delamination; thus, they are not related to a bad interlayer binding. They also stated that the mechanical properties of the zirconia ceramics are affected by porosity and density; they found that the porosities in the 3D-printed zirconia were randomly distributed and they attributed the presence of porosities to the entrapment of air bubbles in the paste of the 3D-printed zirconia.

Fractographic analysis showed similar fracture patterns for the examined specimens of all the tested crowns. The fracture origin was located away from the loading site, in the tensile zone (inner surface of the crowns), and the

direction of crack propagation was outwards; this could be explained by the transverse expansion that occurs in the abutment material owing to the Poisson effect when the crown was subjected to occlusal loading. The Poisson ratio of epoxy (0.31 μ) is similar to that of dentin (0.3 μ) [21]; therefore, the degree of bulging of the epoxy model was expected to be similar to dentin during mastication when subjected to the same occlusal load. The bulging causes the development of hoop stress that causes the fracture [41]. They also showed twist hackles which are formed due to a new stress direction, having the appearance of lances where the small hackle lines merge, as well as arrest lines.

Among the limitations of this study is the use of abutments with no mobility; the dynamic load was vertical with no chewing cycles and in the clinical situation; the crown's design may vary according to the patient. Additionally, the effects of other environmental factors, such as saliva, or the effects of different kinds of beverages and pH fluctuation were not taken into consideration in the present study.

Conclusion

Among the limitations of this in-vitro study, it could be concluded that

1. Zirconia crowns fabricated by the 3D-printing technique can withstand chewing forces for over 4 years (1.2 million cycles).
2. The fracture resistance of these crowns will decrease during function in the oral cavity but is still higher than the masticatory forces (450–520 N).
3. The fracture resistance of the printed crowns is lower than that of the milled crowns but is still higher than the masticatory forces (450–520 N).

Author contribution Conceptualization: AR.

Methodology, investigation, and formal analysis: AR, LG, AF, and LS.

Resources: CB and AR.

Writing original draft preparation: AR.

Writing, review, and editing: CB and LS.

Supervision: CB and LS.

All authors whose names appear on the submission approved the version to be published and agree to be accountable for all aspects of the work in ensuring that questions related to the accuracy or integrity of any part of the work are appropriately investigated and resolved.

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Data Availability Data available on request from the authors.

Declarations

Ethical approval This article does not contain any studies with human participants or animals performed by any of the authors.

Conflict of interest The authors declare no competing interests.

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