Year: 2023

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DOI: https://doi.org/10.1111/ocr.12632

Received: 28 July 2022 | Revised: 27 December 2022 | Accepted: 6 January 2023

DOI: 10.1111/ocr.12632

RESEARCH ARTICLE

Orthodontics & Craniofacial Research WILEY

Fracture toughness and hardness of in-office, 3D-printed ceramic brackets

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Abstract

Objectives: Three-dimensional (3D) printing technology is a promising manufacturing technique for fabricating ceramic brackets. The aim of this research was to assess fundamental mechanical properties of in-office, 3D printed ceramic brackets.

Materials and Methods: 3D-printed zirconia brackets, commercially available polycrystalline alumina ceramic brackets (Clarity, 3M St. Paul, MN) and 3D-printed customized polycrystalline alumina ceramic ones (LightForce™, Burlington, Massachusetts) were included in this study. Seven 3D printed zirconia brackets and equal number of ceramic ones from each manufacturer underwent metallographic grinding and polishing followed by Vickers indentation testing. Hardness (HV) and fracture toughness (K1c) were estimated by measuring impression average diagonal length and crack length, respectively. After descriptive statistics calculation, group differences were analysed with 1 Way ANOVA and Holm Sidak post hoc multiple comparison test at significance level $\alpha = .05$.

Results: Statistically significant differences were found among the materials tested with respect to hardness and fracture toughness. The 3D-printed zirconia proved to be less hard (1261 ± 39 vs 2000 ± 49 vs 1840 ± 38) but more resistant to crack propagation (K1c = 6.62 ± 0.61 vs 5.30 ± 0.48 vs 4.44 ± 0.30 MPa m$^{1/2}$) than the alumina brackets (Clarity and Light Force respectivelty). Significant differences were observed between the 3D printed and the commercially available polycrystalline alumina ceramic brackets but to a lesser extent.

Conclusions: Under the limitations of this study, the 3D printed zirconia bracket tested is characterized by mechanical properties associated with advantageous orthodontic fixed appliances traits regarding clinically relevant parameters.

KEYWORDS
3D printed, ceramic brackets, fracture toughness, hardness, orthodontics, vickers testing, zirconia

1 | INTRODUCTION

Ceramic brackets pose a highly aesthetic fixed appliance option, which is highly popular among adults as well as adolescents.[1–3] The introduction of 3D printing technology seems to provide customized, in-office printed brackets.[4,5] This additive manufacturing method is based on a layer upon layer appliance fabrication thus bypassing the difficulties of the subtractive production techniques. For that purpose, a light source is usually employed capable of polymerizing the light cured photopolymer resin combined with the ceramic powder followed by post heat treatment.[6]

The majority of commercially available ceramic brackets are based on alumina either of monocrystalline or polycrystalline structure.[7] Despite the superior aesthetic properties,[8,9] clinicians are often been confronted with incidences of bracket wing fractures during practice[10,11] since most ceramic materials are strong but at the same time brittle in nature.[7,11] This hinders orthodontic treatment, compromises enamel integrity[12–14] as it complicates the removal process, and increases cost because a new bracket must be placed instead. In an attempt to overcome this inherent property, zirconia based ceramics[15] are attractive candidates as a solution to the issue. Zirconia (ZrO_2) is a material of choice when fracture toughness along with aesthetics are considered,[16,17] owing to its unmatched properties among other ceramics.[18]

The aim of this research is to evaluate fundamental mechanical properties, like fracture toughness and hardness, of a 3D printed zirconia material and compare them to commercially available and customized 3D printed polycrystalline alumina based bracket. The null hypothesis is that there is no significant difference in mechanical properties between the materials tested.

2 | MATERIALS AND METHODS

Seven commercially available ceramic brackets (Clarity, 3M St. Paul, MN) and equal number of 3D printed customized ones (LightForce™ Burlington, Massachusetts) made of polycrystalline alumina and seven 3D printed brackets of zirconia INNI-CERA A2 (AON, Seoul, Korea) comprised the sample. The 3D virtual brackets were exported from the software Ubrackets (Coruo, Limoges, France) and virtually positioned on the Zipro D (AON, Seoul, Korea) zirconia printer's software (Version Zipros, AON, Seoul, Korea) virtual platform. Brackets (Figure 1A) were automatically designed using Ubrackets CAD software (Coruo, Limoges, France) which is software for designing customized orthodontic brackets. Slurry zirconia of particle size ranging from 100 to 900 nm underwent vat-polymerization via digital light processing (DLP). The slurry consisted of zirconia and UV binder with zirconia content of more than 80% and UV binder containing photoinitiator, monomer, oligomer and additives. The printing process was performed under normal atmospheric pressure, at temperature of 25°C and below 70% humidity. The thickness of the wings was 1 mm. Inni-cera slurry contains a Zirconia mixture [(mol%): ZrO_2 92.5%, Al_2O_3 0.07%, SiO_2 0.58%, MgO 0.14%, Na_2O 0.14%, K_2O 0.07%, SnO_2 0.15%, Y_2O_3 4.67%, HfO_2 1.64%] at a percentage ranging between 80~85 (wt%) before and approximately 100% after printing The minimum layer thickness of the material should be 0.7 mm. Printing was done in a 50 μm z axis resolution. The pixel size of the printer's projector is 40 μm and the resolution is 1920 x 1080 pixel (Figure 1).

Printing time was approximately 3 hours with the platform speed movement set at 200 mm/min and printing layer thickness adjusted at 50 μm. After printing the manufactured objects were debinded and sintered in Shenpaz SintraPRO sintering unit for 21 hours and 45 minutes (ShenPaz, MigdalHaemek, Israel) to provide final orthodontic brackets (Figure 1B).

The specimens were embedded in acrylic resin (Verso Cit-2, Struers, Ballerup, Denmark) and the brackets wing surfaces were placed parallel to the horizontal plane. Then, the samples were ground up to 4000 grit SiC paper under water cooling, and were polished with a water-based diamond suspension (NapR1, DiaPro, Struers, Ballerup, Denmark) of up to 1 μm in a grinding/polishing machine (Dap-V, Struers, Ballerup, Denmark). Vickers hardness testing was conducted using a hardness tester machine (Amsler Otto Wolpert-Werke Diatestor 2, Langenfeld, Germany) at ambient temperature. A pilot study was set up to identify the appropriate load for each material employing the criteria of no bracket fracture and detectable cracks around indentation. Alumina brackets were broken with load

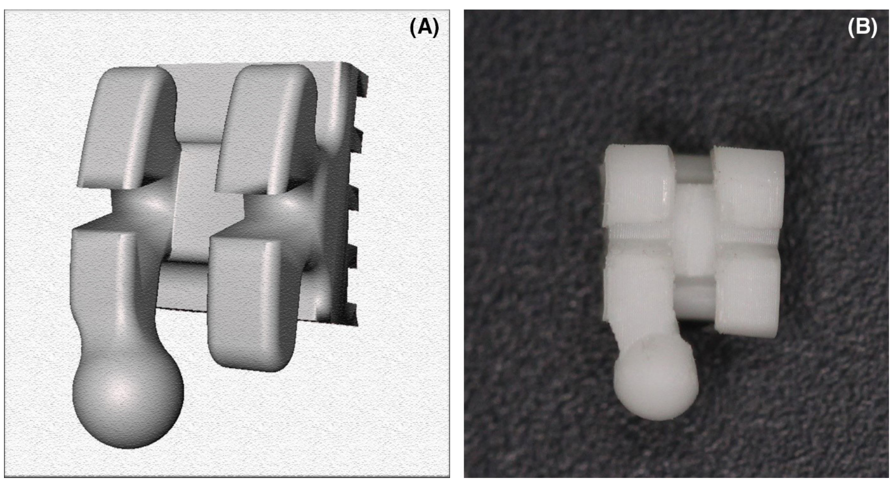

FIGURE 1 Virtual model designed by dedicated software (A) and 3D printed zirconia based orthodontic bracket (B)

above 50 N while at least 200 N were essential to provide detectable cracks around indentation for zirconia (Figure 2). Therefore, the applied force was set at 50 N and 200 N for the alumina and zirconia, respectively. The implementation time was synchronized at 8 seconds. Three indentations were introduced to each specimen, 63 in total. Vickers hardness estimation included the calculation of the diagonal lengths average of the impression left by the indenter (Figure 2B) via the use of an automated digital light microscope (Leica DM4000B, Wetzlar, Germany). Similarly, fracture toughness (K1c) assessment was performed by measuring the crack length originating from the edges of each indentation (Figure 1) and taking into account Vickers hardness and modulus of elasticity according to Lankford's formula.[18]

$$K1c = 0.0782\,(HV\,a^{1/2})\,(E/HV)^{2/5}(c/a)^{-1.56}.$$

Where, HV, Vickers hardness, a, half of the diagonal length, E, Young's modulus, c, crack length. K1c is given in MPa $m^{1/2}$.Modulus of elasticity was set at 210 and 370GPa for zirconia and alumina, respectively.[19]

Descriptive statistics included hardness and fracture toughness mean values and standard deviations. Their significance among three group means was evaluated by one-way ANOVA and Holm Sidak post hoc multiple comparison test, after testing for normality and equal variance with Shapiro–Wilk and Brown-Forsythe test respectively. For all tests a 95% level of significance was set and thus $P < .05$ was predetermined as denoting significant differences.

3 | RESULTS

The results of the mechanical properties tested including their statistical analysis are presented to the Table 1. The 3D printed zirconia showed higher mean values of fracture toughness than the Clarity and LightForce alumina brackets (6.62 vs 5.30, 4.44 MPa $m^{1/2}$, respectively). However, the opposite was true for hardness (1261 vs 2000 and 1840). On the other hand, the Clarity alumina brackets proved to be harder and more resistant to crack propagation than the Light Force. All group mean values differences were statistically significant ($P < .05$).

4 | DISCUSSION

According to the results of this study the null hypothesis cannot be accepted as significant differences were allocated in both properties tested.

Many studies utilize mechanical properties like modulus of elasticity and strength to describe the brittle behaviour of ceramics[10,20–22] and this can lead to misconceptions to the unfamiliar clinician with these terms. Although ceramic brackets exhibit much higher values of the aforementioned parameters than the metallic ones, they are susceptible to fracture and chipping due to their brittle nature.[23] Fracture toughness on the other hand which is the critical value of crack initiation and propagation outlines better the material's resistance to fracture.[23,24] Crack initiation is a process that forms cracks and this phenomenon (as it is anticipated) has been reported for 3D printed zirconia as well.[25] Therefore, it was chosen as the primary judgement tool for wing fracture appraisal.

Fracture toughness of the majority of dental ceramics in a raw form is tested according to ISO24370, which is not applicable for prefabricated small sized specimens such as ceramic brackets.[26] Hence, Vickers indentation testing method was adopted as an alternative for the assessment of mechanical properties like hardness and fracture toughness.[24,27] In case of fracture toughness estimation, Lankford equation was selected since it is applicable not only for the Palmqvist cracks but as well as the median ones.[28] The implemented force was set within the ideal range according to Coric et al.[27] However, the group applied force difference is explained by the fact that lower values than 200 N could not produce distinctive cracks to the zirconia meanwhile alumina brackets had a tendency to fracture when the force exceeded 50 N. Hardness evaluation on the other hand was not affected by force.

Based on the results of this research the 3D printed zirconia proved to be 20% and 30% more resistant to crack propagation than the Clarity alumina and the LightForce 3D printed brackets, respectively. This can be attributed to the well-known zirconia transformation toughening phenomenon where the tetragonal phase shifts to the monoclinic one upon stress in front of crack tip.[28] The resultant increase of volume alters the propagation of crack improving material's fracture resistance. In addition, it is reported that there

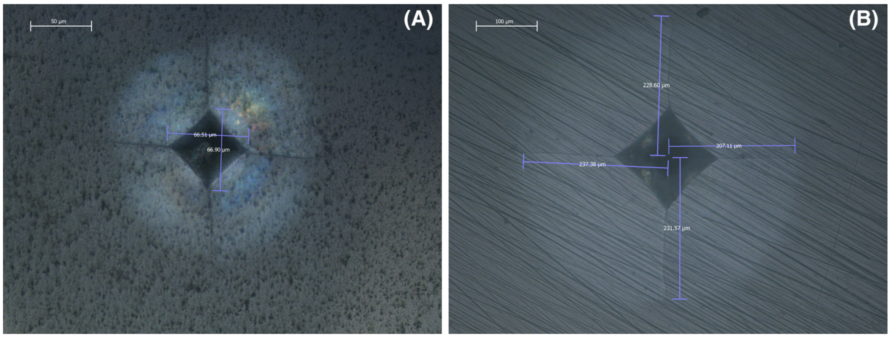

FIGURE 2 Vickers impressions of the alumina brackets (A) and the 3D printed zirconia (B) under the optical microscope at 50x nominal magnification. Crack lengths (four in number) (B) are measured from the center of the pyramidal indentation up to the crack tips. The crack and diagonal lengths (A) are both averaged in order fracture toughness and hardness to be estimated, respectively

TABLE 1 Mean values and standard deviation in parentheses of HV and K1c along with *P* values

Property	3D printed Zirconia	Clarity	Light Force	*P* value
HV	1261 (39)	2000 (49)	1840(38)	<.001
K1c(MPa m$^{1/2}$)	6.62 (0.61)	5.30 (0.48)	4.44(0.30)	<.001[a]

[a]Clarity vs Light Force comparison $P = .017$.

is a switching of zirconia crystal orientation, known as ferroelastic toughening,[29] in front of crack tip further inhibiting crack extension. Alumina brackets on the other hand do not possess such protective mechanisms. Nevertheless, other factors apart from material consistency like porosity and defects[30] in general may have equally important effect on crack propagation. The results of this study are in agreement with previous published data.[27,31] From a clinical point of view, brackets made of zirconia may have an advantageous effect on wing fracture incidence. Consequently, treatment duration is not prolonged and tooth health is not compromised by bracket replacement. Furthermore, fixed appliance fracture during debonding may not be as prominent as in alumina made ceramic brackets.[32,33] However it is noteworthy to be mentioned that the aforemention clinical implications are based on materials property and their true impact in every day practise still to be proved by clinically based studies.

As far as hardness concerned, Clarity alumina bracket and the LightForce one exhibited 37% and 22% higher Vickers value than the 3D- printed zirconia, respectively. The former is comparable to results of previous studies.[7] In contrast to fracture toughness, the materials resistance to plastic deformation is not always beneficial from a clinical perspective. Increased hardness protects the bracket itself from wear and scratching which in turn reduces fracture toughness[34] and more effectively transmits orthodontic archwire forces. However, whatever comes in contact with it rapidly deteriorates. For example, orthodontic archwires which are less hard like those made of NiTi alloy[35,36] are subjected to wear at a faster rate and need to be replaced regularly. The same is true for the stainless steel wires but to a lesser extent.[35,36] On the other hand, the alumina abrasive potential to antagonist teeth becomes clear when the enamel hardness of enamel is considered, which is 8–10 times less than the ceramic brackets.[37] This is a well-known iatrogenic side effect of ceramic brackets in general[38,39] which may be further enhanced when alumina is the material of choice. Patients with increased deep bite and bruxism should not receive alumina brackets in the lower arch since these are known to cause abrasion.[40,41]

In regard to the property differences exhibited by the two types of alumina brackets, these may be attributed to consistency variations and the manufacturing process affecting structure. The majority of commercially available polycrystalline alumina ceramic brackets like Clarity are fabricated via ceramic injection moulding (CIM) technique where the flowable Al_2O_3 particles in combination with the binder are injected to bracket moulds under heat and pressure. In contrast, 3D-printed customized appliances are produced layer upon layer in an additive manner by the use of a laser source sintering the alumina powder. As far as consistency concerned, is self-evident that small deviations in Al_2O_3 powder may further influence the end product behaviour. The Clarity brackets proved to be harder and more resistant to fracture than the LightForce ones which may be associated with the aforementioned clinical traits. Nevertheless, the comparison to the zirconia material seems to be similar for both bracket types.

The present report evaluated fracture toughness and hardness. Further future reports are needed to test other characteristics essential for clinical efficiency and biocompatibility, such as modulus of elasticity, flexural strength, cytotoxicity, adhesive strength and forces generated during in service conditions before the experimental devices can be widely accepted for everyday practice.

The direct clinical implication of the fracture toughness variations deals with the reduced susceptibility of 3D-printed zirconia brackets which might reach a size that could affect its fracture in vivo during treatment especially when torsional moments applied by heavy rectangular archwires; and also during debonding where the reduced brittleness might facilitate a less complex debonding pattern with less fractures of the wings.

5 | CONCLUSIONS

The 3D-printed zirconia showed increased fracture toughness properties compared to polycrystalline alumina brackets and thus can be regarded as a possible candidate for manufacturing customized aesthetic brackets of high resistance to wing fractures.

AUTHOR CONTRIBUTIONS

Conception TE and SZ; production, material preparation, NP; methodology, SZ; specimen preparation, data acquisition, GP; data analysis, interpretation, SP, SZ; drafting, all.

ACKNOWLEDGEMENT

Open access funding provided by Universitat Zurich.

CONFLICT OF INTEREST

The author(s) declared the following potential conflicts of interest with respect to the research, authorship, and/or publication of this article: Nearchos Panayi declared a financial interest with the company Coruo (Limoges, France) concerning the orthodontic computer-aided design software UBrackets, but did not participate in specimen testing or data analysis.

DATA AVAILABILITY STATEMENT

The data that support the findings of this study are available from the corresponding author upon reasonable request.

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How to cite this article: Polychronis G, Papageorgiou SN, Riollo CS, Panayi N, Zinelis S, Eliades T. Fracture toughness and hardness of in-office, 3D-printed ceramic brackets. *Orthod Craniofac Res*. 2023;26:476-480. doi:10.1111/ocr.12632