



Original Article

Strength and Reliability of Fabricate Zirconia by Additive Manufacturing

Hasham Khan¹, Muhammad Amer Khan^{1*}, Shehzad Fahad², Aimen Tariq³, Shawana⁴ and Zainab Ayub⁵¹Department of Science of Dental Materials KMU, Institute of Dental Sciences, Kohat, Pakistan²Royal Imperial Hospital, Swat, Pakistan³Type-D hospital Havelian, Abbotabad, Pakistan⁴Rehman College of Rehabilitation sciences (RCRS), Peshawar, Pakistan⁵Khyber Medical University (KMU), Peshawar, Pakistan

ARTICLE INFO

Key Words:

Additive Manufacturing, Fabricate, Zirconia, Clinical practice, Strength

How to Cite:

Khan, H. ., Amer Khan, M. ., Fahad, S. ., Tariq, A. ., Shawana, ., & Ayub, Z. . (2022). Strength and Reliability of Fabricate Zirconia by Additive Manufacturing: Strength and Reliability of Fabricate Zirconia by Additive Manufacturing. Pakistan Journal of Health Sciences, 3(06).

<https://doi.org/10.54393/pjhs.v3i06.374>

*Corresponding Author:

Muhammad Amer Khan
Department of Science of Dental Materials KMU,
Institute of Dental Sciences, Kohat, Pakistan
amerkhan756@gmail.com

Received Date: 1st November, 2022

Acceptance Date: 25th November, 2022

Published Date: 30th November, 2022

ABSTRACT

Zirconia's distinctive intrinsic qualities have drawn the interest of the dentistry community in medical settings. The technology of additive manufacturing (AM), which produces very little waste, has been utilized to create complex and highly accurate materials. Despite AM has a number of potential benefits for efficiently producing functional, complicated shape zirconia components, there is still a paucity of industrial importance in implementations. **Objective:** To evaluate the strength and reliability of zirconia manufactured using the AM technology. **Methods:** A 3D printer was used to create zirconia bars in both horizontal and vertical orientations. The samples' geometrical correctness, density, layer thickness, and ductility were all measured using short bars. In tests for tensile properties, long bars were utilized. Using a caliper, the lengths of three short bars were measured, and the average values were calculated. They were contrasted with the theoretical parameters using a one-sample t-test. **Results:** It was discovered that varied construction orientations affect dimensional correctness, translucency, and dynamic qualities. Vertical-printed zirconia is denser and translucent than horizontally-printed zirconia. **Conclusions:** Nonetheless, zirconia that has been printed horizontally has remarkable precision and mechanical qualities. Stress and poor adhesion between the layers of materials should be fixed.

INTRODUCTION

The dentistry community has been interested in zirconia ceramics due to its special qualities as an endodontically treated teeth materials and the rising patient desire for attractive, metal-free restorations [1]. Due to their exceptional mechanical characteristics, biocompatibility, high stability, and visual qualities, zirconia restorations are being utilized extensively in dentistry [2, 3]. Additionally, monolithic zirconia restorations becoming increasingly common in hospitals because to greater translucency and improvements in coloring techniques. Zirconia restorations provide additional benefits because of its mechanical characteristics [4], such as requiring less tooth preparation and removing the possibility of veneer

chipping for porcelain fused to zirconia restorations [5]. The term "net shaping forming" is frequently used to describe additive manufacturing, creating the idea that it will automatically generate items with "precisely the same" structure as the suggested conceptual layout. This is false with in majority of instances, especially when drying, debinding, and sintering are involved both before and after printing, such as is the situation with the present ceramics stereolithography process used to produce zirconia dental prosthesis [6]. Additive manufacturing (AM) offers various benefits and has established itself as a legitimate method of producing metals and polymers for use in dentistry [7]. Three-dimensional (3D) printing, also known as AM, which

is different from subtractive manufacturing in that it produces less waste, has been applied to create complex and highly precise materials [8]. Zirconia is one of the dental ceramics that is growing in popularity and application in dentistry because of its exceptional qualities [1, 2, 9]. It provides the opportunity to construct the structure and morphology of ceramic components without geometrical restrictions in a material-saving way since it is the only method for ceramic AM that is now commercially available [10]. Thus, it is advantageous to use AM techniques to considerably lower production waste and associated manufacturing costs [11]. Although AM has a number of potential benefits for efficiently producing functional, complicated shape zirconia components, there is still a paucity of industrial interest in actual applications. The purpose of this study was to evaluate the strength and reliability of fabricate zirconia manufactured using the AM principle.

METHODS

Zirconia powder (Guangdong Orient Zirconic Ind. Sci. Tech. Co., China) and alumina powder (AztroGrit, USA) were combined in a 4:1 weight (wt) ratio. After that, ultrasonography was used to dilute the combination in ethanol. Afterwards, zirconia balls were used to ball-mill the blending in a planetary ball mill for six hours. After that, the mixture was dried for 12 hours in a 60 °C oven. Lastly, a 100-mesh screen was used to sift the dry powder. Four ingredients made up the blended solution that was utilized to make the enameled suspension: acrylamide, N, N' methylenebisacrylamide, glycerin, and deionized water. With a volume concentration of 30 vol%, the powder was then mixed to the mixture. Polyvinyl pyrrolidone K-15 was chosen as 1.2 wt% of the powder, and it was utilized as the dispersion to create the ceramic suspension. After that, zirconia balls were used to ball-mill the ceramic suspension for 12 hours. A vacuum mixer was then used to de-gas the suspension for 30 minutes. Under ultrasound, 2-hydroxy-2-methyl-1-phenyl-1-acetone was added to the ceramic suspension as an initiator in order to create a UV-curable ceramic suspension. The mass fraction of the photo initiator used in this investigation was set to 1 weight percent of the premixed solution. The Magics programme was used to produce the structural framework and slice the sections after the UG application was used to make the 3D model. The stereolithographic machine was then equipped with the final data, which had an x-y resolution, laser beam diameter, and layer thickness of 0.1 mm, 0.06 mm, and 0.07 mm. two distinct patterns of zirconia tiny bars (22 mm long) and long bars (36 mm long) i.e., vertically (V) and horizontally (H) was obtained by stereolithography using the ceramic suspension mentioned above (Figure 1). Both were 3mm

thick and 4mm wide. The short bars were printed (V), lying on three 4 mm² faces and for (H), the long and short bars lying on four 36 mm² faces and four 22 mm² faces respectively. The specimen dimensions' correctness, densities, layer thickness, and flexural strength were all measured using short bars. In tests for toughness, long bars were employed. They went through a binder burning procedure to produce dense parts after being ultrasonically cleaned with ethanol.



Figure 1: Long bars of V and H-group samples: a) shows a V-sample bar and b) shows a H-sample bar

One-way ANOVA was utilized to perform the study, and post hoc was employed to compare the groups for group H. The As-V and Polished-V-samples were compared using independent-samples t-tests. The sets for group H were compared using LSD post hoc, and one-way ANOVA was utilized to conduct the study. The As-V and Polished-V-samples were compared using independent-samples t-tests. The obtained products were sent to the Scanning Electron Microscopy Lab. at Centre for Nanotechnology and Advanced Materials Research, UET, Lahore is fully operational and scanning electron microscopy was used to investigate the fracture surfaces of the short bars following 3-point bend tests. The structural dependability of the AM zirconia fabricate was characterized with Weibull-modulus and Weibull-characteristic strength. Utilizing the methodology covered by Xiang et al., the fracture toughness was evaluated and the comparison was analyzed through LSD post hoc test and one-way ANOVA [11]. The data were normal and the variance was homogeneous. Statistical software (SPSS Statistics for Windows, version 26.0) was used to conduct all statistical analyses.

RESULTS

To explore the stress concentration, that might cause the materials' distortion and loss of accuracy, volumetric shrinking was examined. Only the width of the group H-samples deviates noticeably from the expected dimensions, whilst the length of the group V-samples shows the most disparity (Table 1). The assembling altitude

of the Z-axis layers represented by these two measurements. This specifies that a minor stack height is associated with precise dimension.

Group	Amount	Thick ± SD	Wide ± SD	Lengthy ± SD
V	17	2.94 (±0.02)*	3.97 (±0.09)*	22.24 (±0.10)*
H	16	2.95 (±0.04)*	3.97 (±0.05)*	21.96 (±0.04)*

*P= 0.05 displays statistically relevant variations from the theoretical dimensions

Table 1: Dimensional mean and standard deviations (SDs) in millimeters

With the exception of the thickness of H-samples and length of the V-samples, which are spread fairly uniformly in the bar graphs shown in Figure 2 in which the majority of data is lower in comparison with theoretical value (almost all points are above the theoretical values). This demonstrates that more shrinkage than anticipated happens in the XY direction during the sintering process. The wide fluctuations in size demonstrate the unreliability of the findings for huge diameters. The sole mean value that exceeds the predicted value is the length of V, demonstrating that the shrinkage in the Z axis direction is less severe than expected. Low shrinkage is thought to be a factor in potential deformation and the development of flaws between subsequent layers. When the printing dimensions are huge, this tendency is amplified (22 mm).

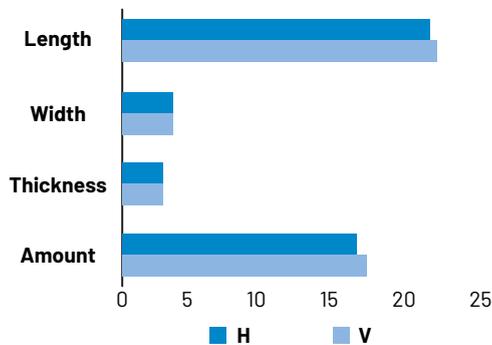


Figure 2: Measurements of the samples bars

The width of H is equal to the theoretical value thanks to a balance struck between the excessive shrinking and layer-by-layer delamination. This suggests that printing accuracy may not match clinical standards, particularly when the stacking height is composed of various thicknesses. Yet, these findings offer a benchmark for raising printing precision. Table 2 shows the initial H-group samples' flexural strength. The results clearly show that the Polished-H-samples have a higher flexural strength. Although stereolithography (SLA) is a near-net shaped process, strengths of components along with surface morphologies in as-sintered SLA ceramic are critical for calculating the dependability of these materials when used in dental restorations.

Prime Groups	Groups	Amount	MPa	Weibull moduli	Weibull characteristics
H	As-H	8	895.43 (±174.96)	6.50	920.22
	Polished-H	6	1095.94 (±200.98)		
V	As-V	17	205.73 (±32.00)	7.99	219.59
	Polished-V	4	226.45 (±46.10)		

Table 2: Characteristics of study samples

The flexural strength between the Polished-V and As-V samples i.e., (226.44 45.10 MPa) and (205.73 32.00 MPa) almost resemble each other. Despite the samples in the V-group having denser samples than those in H-group, an opposite tendency in flexural strength discovered. The samples that were printed vertically had parallel layers to the force applied, which is one explanation for this. This shows that the binding inside a single layer is stronger than the bonding between the layers. The samples manufactured vertically before and after polishing had identical flexural strengths. This behavior further confirms that there is insufficient adhesion between neighboring layers and that the effect of surface characteristics on flexural strength is not readily apparent. Layers are securely sintered into a complete body as evidenced by structural characteristics that have a minor ripple-like appearance. It needs to be recognized that imperfections including pores, agglomerations, and surface faults are unavoidable. These imperfections raise the possibility of a partial loss of AM zirconia's strength. Because of this, zirconia requires polishing, refining, and tinting in order to increase both its flexural strength and dependability as well as its visual and aesthetic qualities. The porosity in HC, the agglomerations, and the surface faults in V are representative flaws of AM zirconia, respectively. Any kind of sample might have one of these three categories of defects. The size of the data dispersion increases with decreasing Weibull modulus. In contrast to As-V, which has values of 219.6 MPa and 8, As-H has a greater Weibull characteristic strength and modulus of 920.22 MPa and 6.50 (Figure 3). This strength typically fits better when defining the strength of ceramic. The data of As-H appear to be more dispersed due to the lower Weibull modulus.

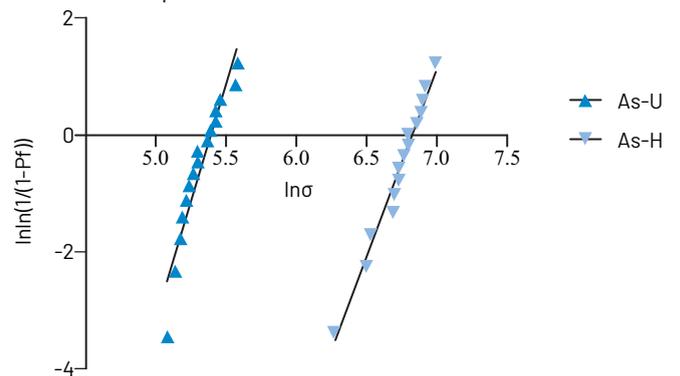


Figure 3: The As-H and As-V-group Weibull plots

The indentation method is widely used to evaluate toughness since it is rapid and simple. In the same test circumstances, this approach may be used to study the difference in fracture toughness of other substances, although the computed results do not truly represent zirconia's fracture resistance. According to ISO 23146, the SEVNB technique is advised for use for testing ceramic durability. The SEVNB technique is not appropriate for zirconia because it is challenging to generate a precise notch-tip radius in yttria tetragonal polycrystalline zirconia; thus, the samples used in this investigation were precisely cut into a V shape. The K_{Ic} value for the H, V, and C groups, which are 12.63 (1.38), 9.29 (1.0), and 14.72 (0.97) MPam^{1/2}, differ noticeably from one another. The H-group can inhibit fracture propagation better than the V-group, as evidenced by the fact that its K_{Ic} values are substantially higher.

DISCUSSION

By adjusting the production parameters, AM has been demonstrated to produce both completely sintered (solid) and partially sintered (more porous) objects. In order to simulate the mechanical characteristics of dentin and enamel and make it possible to create reinforced composite dental restorations, adding pores can alter the material's mechanical properties. Li et al., evaluated the internal and marginal adaptation of zirconia ceramic dental crowns along with the mechanical and physical characteristics of SLA additively generated zirconia crowns [10]. Flexural strengths of 812-128 MPa and a Weibull modulus of 7.44 made the SLA-manufactured zirconia crowns in this experiment strong enough to be used to construct dental crowns. Similar to this, Li et al., investigated and contrasted the efficacy of milled and SLA produced zirconia crowns with chamfer, rounded shoulder, and knife-edge finishing lines [12]. Utilizing 3D deviation evaluation, fabrication reliability was measured, and margin quality was evaluated using optics. Three digital abutment models with knife-edge finishing lines, rounded shoulders, and chamfers of 0.5 mm depth were created. Numerous threshold values could be found in literature for the roughness of surface which obstructs bacterial adherence [13-15]. Because of this, the R_a values of three-varieties of AM zirconia, together with H-coatings with or without manual flaws and surface of V, are still too high even when compared to the roughness value that is considered to be the most acceptable (R_a 0.59 μ m) (the minimum R_a is 0.70 μ m). As a result, if these fabrics were to be utilized in dental restoration, the chance of secondary caries and periodontium inflammation would simultaneously increase. The opposing enamel is also worn down by a rough surface [15, 16]. To prevent the growth of microorganisms and antagonist wear, it is crucial that the

AM zirconia be polished. In the current study, there was no statistically significant difference between the As-H sample and the AM zirconia flexural strength, however both H-samples' strengths were decreases in comparison to the Polished-H sample. The H-group has considerably higher K_{Ic} values for fracture toughness than the V-group, representing that the H-group is enhanced and able to prevent crack propagation. The V-notch and force line are parallel to the layers in the V-group of samples but perpendicular to the layers in the H-group of samples, which may be why specimens with different orientations exhibit varying flexural strengths. Similar findings were also discovered by Xiang et al., [11]. Khanlar et al., analyzed the many AM techniques that may be used to create zirconia, including SLA, direct light processing, direct inkjet printing, and lithography-based (LCM) processes [2]. Only in the SLA among various AM processes was create zirconia found to be strong and reliable. These findings concur with the present research's findings of Ferrage et al., and Lüchtenborg et al., as well [17, 18]. The early lab investigations demonstrate several AM processing methods for zirconia for a variety of clinical uses, mostly in implant and restorative dentistry [19, 20]. Although each method has significant benefits, AM of zirconia for dental applications seems to most frequently use vat polymerization [21]. Although in-vitro investigations indicate that this new technique has similar mechanical qualities and precision to milling and its potential is highly promising, more advancements are required in a number of areas, including printer improvement, material research, and optimizing the printing settings.

CONCLUSIONS

It was discovered that varied construction orientation affects dimensional correctness, translucency, and mechanical qualities. Vertically printed zirconia is denser and translucent than horizontally printed zirconia. Furthermore, zirconia that has been printed horizontally has remarkable precision and mechanical qualities. Tension and a lack of strong adhesion between the materials' successive layers are the main issues that need to be fixed. AM zirconia having considerable promise for use in dental applications and can be employed in single-unit dental prostheses, but more research is required to demonstrate their dependability under conditions that more closely resemble real-world clinical settings.

Conflicts of Interest

The authors declare no conflict of interest

Source of Funding

The author(s) received no financial support for the research, authorship and/or publication of this article

REFERENCES

- [1] Popp C and Zarzar AA. Immunologically Neutral Dental Implants Ceramic (Zirconia) Implants. *Journal of Dentistry and Oral Sciences*. 2022 Sep; 4(3): 1-22. doi: 10.37191/Mapsci-2582-3736-4(3)-137.
- [2] Khanlar LN, Salazar Rios A, Tahmaseb A, Zandinejad A. Additive manufacturing of zirconia ceramic and its application in clinical dentistry: a review. *Dentistry Journal*. 2021 Sep; 9(9): 104. doi: 10.3390/dj9090104.
- [3] Gautam C, Joyner J, Gautam A, Rao J, Vajtai R. Zirconia based dental ceramics: structure, mechanical properties, biocompatibility and applications. *Dalton transactions*. 2016 Nov; 45(48): 19194-215. doi: 10.1039/C6DT03484E.
- [4] Skorulska A, Piszko P, Rybak Z, Szymonowicz M, Dobrzyński M. Review on polymer, ceramic and composite materials for cad/cam indirect restorations in dentistry—Application, mechanical characteristics and comparison. *Materials*. 2021 Mar; 14(7): 1592. doi: 10.3390/ma14071592.
- [5] Manicone PF, Iommetti PR, Raffaelli L. An overview of zirconia ceramics: basic properties and clinical applications. *Journal of dentistry*. 2007 Nov; 35(11): 819-26. doi: 10.1016/j.jdent.2007.07.008.
- [6] Li Y, Lin X, Hu Y, Kang N, Gao X, Dong H, et al. Zirconium modified Nb-22Ti-16Si alloys fabricated by laser additive manufacturing: microstructure and fracture toughness. *Journal of Alloys and Compounds*. 2019 Apr; 783: 66-76. doi: 10.1016/j.jallcom.2018.12.280.
- [7] Bhargav A, Sanjairaj V, Rosa V, Feng LW, Fuh YHJ. Applications of additive manufacturing in dentistry: A review. *Journal of Biomedical Materials Research Part B: Applied Biomaterials*. 2018 Jul; 106(5): 2058-64. doi: 10.1002/jbm.b.33961.
- [8] Sun J, Chen X, Wade-Zhu J, Binner J, Bai J. A comprehensive study of dense zirconia components fabricated by additive manufacturing. *Additive Manufacturing*. 2021 Jul; 43: 101994. doi: 10.1016/j.addma.2021.101994.
- [9] Grech J and Antunes E. Zirconia in dental prosthetics: A literature review. *Journal of Materials Research and Technology*. 2019 Sep; 8(5): 4956-64. doi: 10.1016/j.jmrt.2019.06.043.
- [10] Li R, Wang Y, Hu M, Wang Y, Xv Y, Liu Y, et al. Strength and adaptation of stereolithography-fabricated zirconia dental crowns: an in vitro study. *The International Journal of Prosthodontics*. 2019 Sep; 32(5): 439-43. doi: 10.11607/ijp.6262.
- [11] Xiang D, Xu Y, Bai W, Lin H. Dental zirconia fabricated by stereolithography: Accuracy, translucency and mechanical properties in different build orientations. *Ceramics International*. 2021 Oct; 47(20): 28837-47. doi: 10.1016/j.ceramint.2021.07.044.
- [12] Li R, Chen H, Wang Y, Sun Y. Performance of stereolithography and milling in fabricating monolithic zirconia crowns with different finish line designs. *Journal of the mechanical behavior of biomedical materials*. 2021 Mar; 115: 104255. doi: 10.1016/j.jmbbm.2020.104255.
- [13] Zhang M, Zhang Z, Ding N, Zheng D. Effect of airborne-particle abrasion of presintered zirconia on surface roughness and bacterial adhesion. *The journal of prosthetic dentistry*. 2015 May; 113(5): 448-52. doi: 10.1016/j.prosdent.2014.12.012.
- [14] Lu A, Gao Y, Jin T, Luo X, Zeng Q, Shang Z. Effects of surface roughness and texture on the bacterial adhesion on the bearing surface of bio-ceramic joint implants: An in vitro study. *Ceramics International*. 2020 Apr; 46(5): 6550-9. doi: 10.1016/j.ceramint.2019.11.139.
- [15] Özarıslan M, Bilgili Can D, Avcioglu NH, Çalışkan S. Effect of different polishing techniques on surface properties and bacterial adhesion on resin-ceramic CAD/CAM materials. *Clinical Oral Investigations*. 2022 Apr; 26: 5289-99. doi: 10.1007/s00784-022-04497-8.
- [16] Yu P, Wang C, Zhou J, Jiang L, Xue J, Li W. Influence of surface properties on adhesion forces and attachment of *Streptococcus mutans* to zirconia in vitro. *BioMed Research International*. 2016 Nov; 2016: 8901253. doi: 10.1155/2016/8901253.
- [17] Ferrage L, Bertrand G, Lenormand P, Grossin D, Ben-Nissan B. A review of the additive manufacturing (3DP) of bioceramics: Alumina, zirconia (PSZ) and hydroxyapatite. *Journal of the Australian Ceramic Society*. 2017 Apr; 53(1): 11-20. doi: 10.1007/s41779-016-0003-9.
- [18] Lüchtenborg J, Willems E, Zhang F, Wesemann C, Weiss F, Nold J, et al. Accuracy of additively manufactured zirconia four-unit fixed dental prostheses fabricated by stereolithography, digital light processing and material jetting compared with subtractive manufacturing. *Dental Materials*. 2022 Sep; 38(9): 1459-69. doi: 10.1016/j.dental.2022.06.026.
- [19] Tachibana K, Atsuta I, Tsukiyama Y, Kuwatsuru R, Morita T, Yoshimatsu H, et al. The need for polishing and occlusal adjustment of zirconia prostheses for wear on antagonist teeth. *Dental Materials Journal*. 2021 May; 40(3): 650-6. doi: 10.4012/dmj.2020-190.
- [20] Zarone F, Russo S, Sorrentino R. From porcelain-fused-to-metal to zirconia: clinical and experimental considerations. *Dental materials*. 2011 Jan; 27(1): 83-

96. doi: 10.1016/j.dental.2010.10.024.

- [21] Galante R, Figueiredo-Pina CG, Serro AP. Additive manufacturing of ceramics for dental applications: A review. *Dental materials*. 2019 Jun; 35(6): 825-46. doi: 10.1016/j.dental.2019.02.026.