

The effect of low-temperature degradation and building directions on the mechanical properties of additive-manufactured zirconia

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This study aimed to investigate the effect of low-temperature degradation (LTD) on the mechanical properties of additive-manufactured zirconia. In addition, the mechanical properties of additive-manufactured were compared with those before aging under similar experimental conditions. This study prepared stereolithography apparatus fabricated zirconia specimens with flexural strength, modulus of elasticity, Vickers hardness, and fracture toughness. The specimen position data were set as parallel (0°), diagonal (45°), and perpendicular (90°) to the direction of the building. The LTD condition was 5 h under 134°C and 0.2 MPa in an autoclave. It was found that the 0° direction differed significantly from all other conditions before and after aging, and the highest flexural strength was obtained when the additive specimen was manufactured perpendicular to the building direction. However, the results indicate that there is a negligible effect of aging on the mechanical properties of additive-manufactured zirconia.

Keywords: Aging, Anisotropy, Digital dentistry, Y-TZP, 3D-printing

INTRODUCTION

Polycrystalline yttria-stabilized tetragonal zirconia (Y-TZP) is the most commonly used zirconia among dental ceramics, as it has the highest strength^{1,2}. Conventional Y-TZP is often used in the fabrication of porcelain veneers due to its high opacity and for esthetic purpose, and several long-term clinical reports have been published on this topic³⁻⁵. Improvements made in zirconia in recent years have enabled the clinical use of the monolithic structure without porcelain veneering, and clinical reports published on this topic have been increasing^{6,7}. Y-TZP containing 3 mol% yttria and approximately 0.25 wt% alumina is used as a framework in dentistry. However, Y-TZP degrades after long-term use in high-humidity and temperature environments such as the oral cavity^{8,9}. There is an observed phase transformation where tetragonal crystals transform to monoclinic crystals, and additionally a 4% volume expansion when stored in humid conditions for long periods, even in low-temperature environment, such as the oral cavity¹⁰. Low-temperature degradation (LTD) is the transformation of a portion of the tetragonal phase of zirconia on the material surface into the monoclinic phase when exposed to water or moisture at low temperatures, resulting in the formation of microcracks and increased surface roughness¹¹. Since this process results in a reduction in the fracture strength of the material¹²,

the international standards for zirconia implants have formulated a test to evaluate the accelerated aging, and specifies that the percentage of monoclinic crystals must be less than 25% after steam sterilization at 134°C and 2 atm for 5 h and the strength loss after the test must be less than 20%. Products that meet these requirements are commercially available, eliminating concerns regarding accelerated aging. However, this can be a significant issue for zirconia restorations with monolithic structures because of their direct exposure to saliva and occlusal loading, which promotes LTD.

Dental computer-aided design/computer-aided manufacturing (CAD/CAM) systems are commonly used in clinical practice and are based on the principle of subtractive manufacturing. In recent years, CAD/CAM systems with lesser material wastage and additive manufacturing, which create a form while layering materials, have been introduced. The development of additive manufacturing technologies and stereolithographic techniques, such as stereolithography apparatus (SLA) and digital light processing (DLP), have demonstrated promising results regarding the fabrication of zirconia-based dental prostheses¹³. These techniques enable the layer-by-layer printing of three-dimensional (3D) objects by curing a photocurable resin within a zirconia paste. This process is followed by post-treatment processes, such as binder removal and sintering, which are necessary to remove the

photocurable resin and produce a dense material¹⁴. Nevertheless, although these technologies have enabled the rapid printing and production of complex geometries with high precision and excellent surface quality^{13,15}, there are concerns regarding the accuracy of zirconia-printed models due to the presence of the photocurable resin and the degreasing process.

The mechanical properties of additive-manufactured zirconia are affected by anisotropy in the building direction, which impacts flexural strength^{16,17}. Depending on the building direction, a flexural strength of 800 MPa or higher and sufficient strength can be obtained^{17,18}. However, the relationship between LTD, the characteristics of zirconia, and anisotropy in the building direction remains to be clarified. Thus, this study investigated the effects of LTD on the mechanical properties of additive-manufactured zirconia. In addition, the mechanical properties before and after LTD under similar experimental conditions were compared.

MATERIALS AND METHODS

The SLA-fabricated zirconia specimens were prepared with normalized flexural strength, modulus of elasticity, Vickers hardness, and fracture toughness. Characterization was performed to determine the associations between the mechanical properties of the SLA-fabricated zirconia specimens, the three architectural orientations, and aging. The specimen position data were set as parallel (0°), diagonal (45°), and perpendicular (90°) to the direction of the building (Fig. 1). For the evaluation of flexural strength and elastic modulus, two distinct specimens were fabricated for the 90° direction: one in the X-axis direction, horizontal to the building phase (90°-x), and one in the Y-axis direction, vertical to the building direction (90°-y).

The digital designs of the specimens were created using open-source software (3D Builder, v.18.0.1931.0, Microsoft, Redmond, WA, USA) and saved in standard tessellation language (STL) format. The specimens

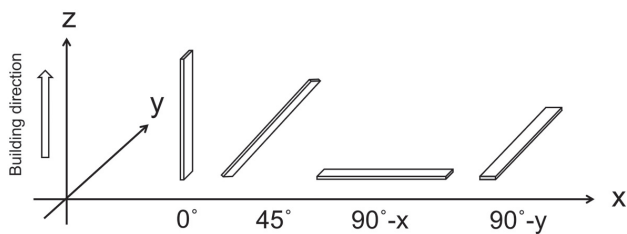


Fig. 1 Direction of construction of the additive-manufactured zirconia specimens with an example of the specimens used in the flexural strength test.

The specimens are set parallel (0°), diagonal (45°), and perpendicular (90°) to the building direction. In the 90° direction group, the specimens have the X-axis horizontal to the building stage (90°-x) and the Y-axis perpendicular to the building direction (90°-y).

were fabricated using the SLA method with liquid bath photopolymerization (CeraMaker 900, 3DCeram, Limoges, France) and a dedicated ZrO₂ (zirconia stabilized with 3 mol% yttria) paste (3DMix ZrO₂, 3DCeram) as the polymerization material. The ZrO₂ paste was placed in the bath and polymerized one layer at a time. A computer-controlled laser beam irradiated the slurry surface in a pattern determined by the shape of the object¹⁹, leading to the solidification and adherence of the first layer of the irradiated resin to the modeling platform²⁰. The laser source was positioned above the tank, and modeling was performed upward. The modeling platform was immersed in the tank, and only a thin layer of resin located just below the resin surface was exposed to the laser source. The modeling table was moved downward along the Z-axis by a thickness of one layer as the layers were cured, and a new resin layer was laid over the cured layer²¹. Thus, a 3D object was constructed by repeatedly moving and curing the platform. After printing, the green portions were placed in a debinding furnace to remove the organic matter and excess resin *via* debinding. The resulting ZrO₂ was degreased using a solvent, dried, and sintered^{14,22} in a furnace at approximately 1,500°C. Details of the sintering process are provided by the manufacturer with proprietary information. Since ZrO₂ shrinks by approximately 20% after sintering, the specimens were designed to account for dimensional changes after sintering. All specimens were prepared by the manufacturer.

The model proposed by ISO 13356: 2008²³ was utilized for aging the zirconia specimens, with the aging time set as 5 h under the conditions of 134°C and 0.2 MPa in an autoclave (PC-242HS, Hirayama, Saitama, Japan).

Flexural strength of zirconia specimens was evaluated according to ISO 6872: 2015²⁴. Specimens with the dimensions 1.2×4.0×25.0 mm were prepared and then polished with #400 and #800 water-resistant abrasive paper (Struers, Copenhagen, Denmark) (*n*=10). The fabricated specimens were immersed in ultrapure water (Milli-Q Reference, Merck, Burlington, MA, USA) at 37°C for 24 h and then underwent three-point bending tests in a universal testing machine (AGS-X, Shimadzu, Kyoto, Japan) with a 12 mm distance between the fulcrum points and a 1.0 mm/min crosshead speed. The stress-strain curve derived from the test was used to determine the slope of the linear portion. The flexural strength σ (MPa) is expressed as follows:

$$\sigma = 3PL/2wt^2,$$

where *P* is load at failure; *L* is 20 mm distance between the supports; *w* is pre-measurement specimen width; and *t* is pre-measurement specimen thickness.

The slope of the linear portion was determined from the stress-strain curves obtained from the tests to calculate the flexural modulus.

Specimens of dimensions 10×10×3 mm fabricated with #400 water-resistant abrasive paper (Struers) were used for the Vickers hardness and fracture toughness tests. The specimens were polished with #800 water-resistant abrasive paper (Struers) and immersed in

ultrapure water (Milli-Q Reference, Merck) for 24 h at 37°C. A Vickers hardness tester (AK15, Akashi, Tokyo, Japan) was used to calculate Vickers hardness. The Vickers indenter was pressed against the surface of the test specimen to generate semicircular or semielliptical vertical cracks around the indentation under a loading time of 10 s at a load of 20 kgf, according to the method outlined by Niihara *et al.*²⁵⁾. The lengths of these cracks were measured, and fracture toughness for the measured values (MPam^{1/2}) was calculated as follows:

$$KIC = 0.203 (c/a)^{-3/2} H a^{1/2},$$

where *KIC* is fracture toughness (MPam^{1/2}), *a* is one-half of the diagonal length of the indentation (μm), *c* is one-half of the crack length (μm), and *H* is Vickers hardness. The fracture toughness was calculated from the results of the Vickers hardness test according to the method outlined by Harada *et al.*²⁶⁾.

The microstructures of the specimens were observed using a scanning electron microscope (SEM; JSM-IT200, JEOL, Tokyo, Japan) at an accelerating voltage of 15.0 kV after sputter-coating each specimen with platinum in an argon gas environment using a sputter-coater machine (E-1030, Hitachi, Tokyo, Japan).

The normality of the data distribution and homogeneity of variance for the measured flexural strength, elastic modulus, Vickers hardness, and fracture toughness were verified using the Shapiro–Wilk test and Levene’s test, respectively. When the results of Levene’s test revealed the variances were equal, both two-way analysis of variance and Tukey’s honestly significance difference test were performed. When the results of Levene’s test did not show homogeneity, nonparametric procedures, including the Kruskal–Wallis test and Steel–Dwass multiple comparison test, were utilized. The significance level was set at 5%, and all statistics were analyzed using SPSS statistical software (IBM SPSS Statistics 24, IBM, Armonk, NY, USA). In the statistical process, parametric tests were selected when all results showed normal and homogeneous distributions.

RESULTS

In the flexural strength tests, the values after LTD tended to be lower than those before LTD. However, there were no significant differences observed before and after LTD in the same building directions ($p > 0.05$) (Fig. 2). It was found that the 0° direction differed significantly from all other conditions before and after LTD, and the highest flexural strength was obtained when the additive was manufactured perpendicular to the building direction.

The elastic modulus after LTD tended to be slightly higher than that observed before aging. The elastic modulus after LTD tended to increase under all conditions, unlike the flexural strength results, there were no significant differences observed before and after LTD in the same building directions ($p > 0.05$) (Fig. 3). The Vickers hardness values showed almost no change before and after LTD, and there were no significant differences observed among the building directions ($p > 0.05$) (Fig. 4). The fracture toughness values ranged from 6.3 to 6.9.

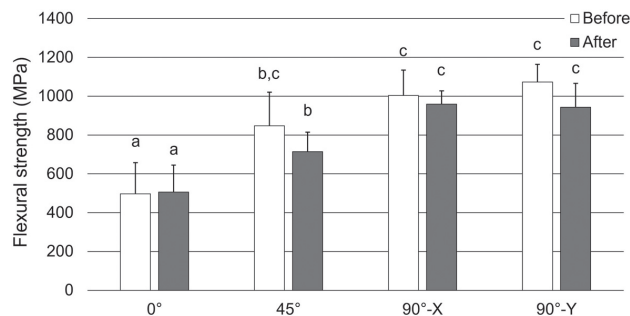


Fig. 2 Flexural strength of the additive-manufactured zirconia specimens before and after LTD. 0°: parallel, 45°: diagonal, 90°: perpendicular. No significant differences are observed between the same lowercase letters. The data before aging are taken from Miura *et al.*¹⁷⁾.

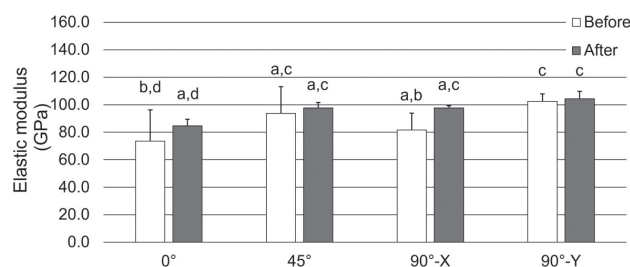


Fig. 3 Elastic modulus of the additive-manufactured zirconia specimens before and after LTD. 0°: parallel, 45°: diagonal, 90°: perpendicular. No significant differences are observed between the same lowercase letters. The data before aging are taken from the study by Miura *et al.*¹⁷⁾.

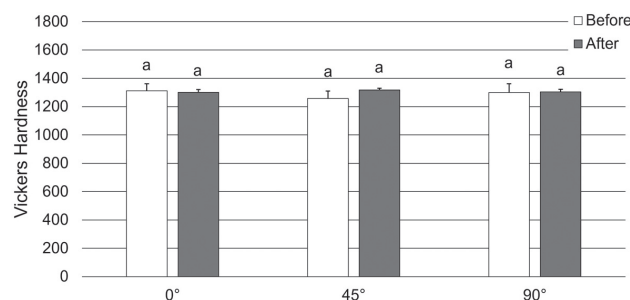


Fig. 4 Vickers hardness of additive-manufactured zirconia specimens before and after LTD. 0°: parallel, 45°: diagonal, 90°: perpendicular. No significant differences are observed between the same lowercase letters. The data before aging are taken from the study by Miura *et al.*¹⁷⁾.

There were no significant differences observed among the building directions ($p > 0.05$). However, before LTD in the 0° direction, significant differences were found both before and after LTD in the 45° and 90° direction (Fig. 5).

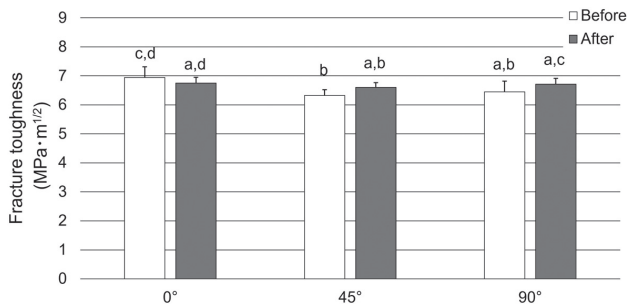


Fig. 5 Fracture toughness of additive-manufactured zirconia specimens before and after LTD. 0°: parallel, 45°: diagonal, 90°: perpendicular. No significant differences are observed between the same lowercase letters. The data before aging are taken from Miura *et al.*¹⁷.

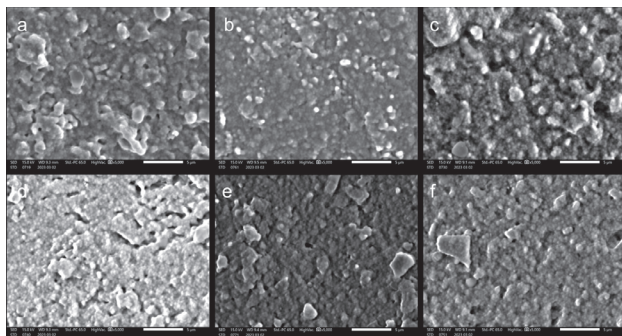


Fig. 6 Scanning electron microscopy images of 0° direction (a, d), 45° direction (b, e), and 90° direction (c, f) specimens at 5000× magnification. Top: Control (a–c), Bottom: After aging (d–f).

SEM micrographs at 5,000× magnification showing the microstructures of the additive-manufactured zirconia specimens before and after LTD are presented in Fig. 6. The SEM image did not reveal an increase in particle size or the appearance of pores before and after LTD.

DISCUSSION

The current study investigated the effects of aging and building direction on the mechanical properties of additively manufactured zirconia. The experimental results demonstrate that there is only negligible effect of aging on the mechanical properties of additively-manufactured zirconia. An accelerated aging test was employed to estimate the zirconia phase transformation *in vivo*, with 1 h of autoclaving at 134°C assumed to be analogous to 3–4 years at 37°C *in vivo*^{10,27}. Using this estimate, 5 h of autoclaving would result in aging equivalent to 15–20 years at 37°C. A similar effect is theoretically applied to a restoration in the oral cavity for 15–20 years, which is an acceptable lifespan for fixed partial dentures (FPDs)²⁸. The test does not

represent actual clinical conditions and therefore may underestimate the degradation of zirconia ceramics in the oral environment²⁹; however, the test is an efficient method for estimation of the materials long-term performance is the most widely accepted method to simulate clinical conditions³⁰, following the International Organization for Standardization guidelines²³. Other aging tests include boiling in hot water or artificial saliva, thermocycling, mechanical cyclic loading, or a combination of thermal and mechanical fatigue²⁵. Ban stated that it is not appropriate to consider the aging of zirconia as a drawback of dental zirconia and that the aging test of zirconia should be considered as an accelerated test of the degradation phenomenon⁹.

In the current study, flexural strength before aging was 800 MPa or higher in the 45° and 90° directions. A material with a flexural strength of 800 MPa can be used as a framework for FPDs with four or more units²⁴. Although the flexural strength in the 45° direction after aging decreased, it still exceeded 500 MPa. Therefore, it can be used in the fabrication of three-unit FPDs²⁴. Previous studies have reported that autoclave aging has no significant effect on strength^{31,32}, and that the strength decreases significantly depending on the processing time, product, and composition of the zirconia system investigated^{31,33–35}. Flinn *et al.* reported that flexural strength decreases following 5–200 h of aging at 134°C and 0.2 MPa³¹. Siarampi *et al.* reported contrasting results which demonstrated that 5 h of aging increased flexural strength, but 10 h decreased flexural strength of zirconia³⁶. The differences in these findings are presumed to be due to differences in aging temperature, as Siarampi *et al.* utilized a lower aging treatment temperature of 121°C (0.2 MPa remained the same). Furthermore, the flexural strength of 3Y-TZP additively-manufactured zirconia fabricated using DLP processing does not differ significantly before and after thermal cycle aging³⁷. Additionally, flexural strength tests using additively-manufactured zirconia fabricated using SLA processing showed that the aging procedure resulted in greater flexural strength decreases in the subtractive manufacturing group than that in the additive manufacturing group. This may be attributable to differences in manufacturing procedures, porosity, chemical composition, and sintering shrinkage¹⁹.

Harada *et al.* reported that 3Y-TZP aged under conditions of 134°C and 0.2 MPa for 5 h showed no significant differences in Vickers hardness and fracture toughness³⁸. Although the test methods were different, 3Y-TZP and aging did not have a significant impact on biaxial flexural strength, indentation modulus, or Marten's hardness; under similar aging conditions³⁹. Furthermore, three-point bending strength, fracture toughness, and Vickers hardness of 3Y-TZP decreased slightly after aging for 20 h⁴⁰. The Vickers hardness of 3Y-TZP has been reported to decrease with autoclaving time exceeding 100 h⁴¹. After 100 h of autoclave aging, nanoindentation testing revealed that the values of hardness and elastic modulus decreased, and that there were significant differences between the two groups

before and after aging⁴²). In the current study, autoclave aging was performed for 5 h and did not affect flexural strength, elastic modulus, Vickers hardness, or fracture toughness; however, it is possible that extending the aging time may affect these physical properties.

The autoclave aging effects on zirconia stability (t-m transformation) depend on temperature, aging time, and type of Y-TZP material. t-m transformation has been reported to cause various changes in mechanical and surface properties, such as grain pull-out and microcracking^{33,43,44}. Phase changes due to LTD are the greatest at 200°C–300°C on zirconia surfaces⁹, which are originally composed of densely sintered tetragonal grains that undergo transformation to monoclinic grains, involving a 3%–5% increase in the grain volume, thereby creating micro-defects on the surface. Water is then able to penetrate into the intergranular defects causing phase transformation to progress into the subsurface. In addition to an increase in surface micro-roughness, a decrease in hardness was also observed. Moreover, there is a degradation of the mechanical strength of the material with the progression of LTD. In addition, the microstructure and chemical and crystalline composition of zirconia define its susceptibility to LTD; larger tetragonal grains are critically susceptible to LTD, whereas cubic-containing zirconia is more stable^{11,45,46}. Furthermore, CAD/CAM-milled materials are reportedly susceptible to subsequent degradation⁴⁷; however, the impact is likely to be low for additive-manufactured zirconia.

This study demonstrates that aging additively manufactured zirconia has little impact on its mechanical properties. The results support the clinical utilization of additive-manufactured zirconia, although it is inferior to subtractive zirconia^{1,2}. Further optimization of the 3D printing procedure might improve the internal material structure, thereby further increasing material reliability. In the future, this technology will become more clinically relevant due to reduced material and equipment costs and improved accuracy. Clinicians may consider using additive-manufactured zirconia, which has shown less LTD after accelerated aging, for crowns and framework for three unit-FPDs.

CONCLUSION

High average material strengths that exceed the current ISO requirements for fixed ceramic prostheses were measured in flexural tests, except in the 0° direction. LTD had little effect on flexural strength, elastic modulus, Vickers hardness, and fracture toughness. However, the optimization of all processing steps, including 3D printing, cleaning, stripping, sintering, and color immersion, is necessary to achieve optimal reliability of 3D-printed zirconia materials for clinical applications.

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