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Original article

The effect of nonthermal argon plasma surface treatment on the fracture resistance of monolithic zirconia restorations containing tetragonal and cubic grains

Hatice Nalan Bozogullari[1)], Hande Citir Yucel[1)], Ozlem Ustun[2)], Tamer Akan[3)], and Fusun Ozer[4)]

[1)] Department of Prosthodontics, Faculty of Dentistry, Alanya Alaaddin Keykubat University, Antalya, Turkey
[2)] Department of Prosthodontics, Faculty of Dentistry, Akdeniz University, Antalya, Turkey
[3)] Department of Physics, Faculty of Science and Letters, Eskisehir Osmangazi University, Eskisehir, Turkey
[4)] Department of Preventive and Restorative Sciences, University of Pennsylvania School of Dental Medicine, Philadelphia, PA, USA

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Abstract

Purpose: The aim of this study was to investigate the effect of nonthermal argon plasma (NP) surface treatment on the fracture resistance of monolithic zirconia restorations with different microstructures.
Methods: Twenty restorations were prepared from each of two tetragonal and two cubic zirconia materials (80 restorations in total). The restorations were then divided into two subgroups ($n = 10$) for each material according to the surface treatment applied: air abrasion or NP. The surface topography of the treated groups was examined using a scanning electron microscope. All restorations were fixed to metal dies with resin cement, subjected to thermal cycling, and then underwent fracture resistance testing with a universal testing device. Two-way ANOVA and Bonferroni tests were used for statistical analysis of the data ($\alpha = 0.05$).
Results: The type of surface treatment and the type of zirconia material were shown to significantly affect the fracture resistance of the restorations. The air-abraded groups showed significantly higher fracture resistance (N) than the NP groups ($P < 0.001$).
Conclusion: The results of this study suggest that air abrasion surface treatment has a more favorable effect on the fracture resistance of tetragonal and cubic zirconia restorations than NP surface treatment.

Keywords: air abrasion, cubic zirconia, monolithic zirconia, nonthermal argon plasma, tetragonal zirconia

Introduction

Among various dental ceramics, zirconia restorative materials have superior mechanical properties, biocompatibility, chemical stability, appropriate marginal adaptation, and esthetics, making them a very popular material in dentistry [1]. Zirconia exists in three crystallographic phases: a monoclinic phase (m) between room temperature and 1,170°C, a tetragonal phase (t) between 1,170°C and 2,370°C, and a cubic phase (c) above 2,370°C. Among these three phases, the tetragonal phase shows excellent mechanical properties at room temperature. Oxides such as CaO, MgO, Ce_2, and yttrium oxide (Y_2O_3) are added to stabilize zirconia in the tetragonal phase. Yttria-stabilized tetragonal zirconia polycrystal (Y-TZP), the most widely used among zirconia materials, is obtained by addition of 3 mol% Y_2O_3 [2]. Of all the ceramic materials available today, conventional tetragonal zirconia polycrystal (3Y-TZP) stabilized with yttrium has the highest fracture resistance due to phase transformation toughening and is used as a core ceramic in all-ceramic restorations in place of metal substructures [3]. Local factors such as stress and surface treatment may also trigger a tetragonal-to-monoclinic (t→m) phase transformation in 3Y-TZP zirconia ceramics. This phase transformation leads to an increase in volume, which slows and stops crack propagation by generating compressive stress and enhances the durability of zirconia [2,4]. However, attaining esthetic restorations with these materials is difficult due to the high opacity of 3Y-TZP. Therefore, the 3Y-TZP ceramic core structure is covered with feldspathic porcelain to satisfy the esthetic expectations of patients. Chipping is the most common complication of these veneer ceramics. In recent years, translucent monolithic zirconia has been produced to improve the optical properties of 3Y-TZP, solve the chipping problem, prevent the removal of excess material from the tooth, and satisfy esthetic requirements [5,6].

Conventional tetragonal zirconia (3Y-TZP) contains 0.25% alumina, whereas second-generation translucent monolithic zirconia (TZ), with improved esthetic properties, contains less than 0.05% alumina by weight [7]. Other factors affecting the translucency of zirconia are microstructural modifications, the amount and type of additives, chemical composition, sintering, grain size, atmospheric conditions during sintering, and cubic phase [5,8,9]. An increase in the yttrium content causes an increase of the cubic phase and the cubic grains show greater volume than tetragonal grains in the structure of the material [5]. The molar percentage (mol%) of yttrium oxide stabilizers in this zirconia is more than 3%. These materials are called third-generation high-translucent partially stabilized cubic containing monolithic zirconia (CZ). With this change in its content, the microstructure of the ceramic crystal comprises approximately 50% cubic phase [9]. However, CZ shows reduced t-m phase transformation due to its more stable cubic form [10], which causes a decrease in mechanical properties. However, it is claimed that the resistance of CZ to low-temperature degradation (LTD) is also increased [11].

Adhesion is an important factor for the long-term clinical success of zirconia restorations. Resin cements are preferred for the cementation of zirconia because of their excellent retention, marginal fit, and the durability they impart to the restoration [12]. However, the low surface energy of zirconia decreases wettability. On account of this, suitable surface treatments are required for better adhesion between the zirconia surface and the resin cement. Many different methods of surface treatment are available, such as tribochemical silica coating, laser treatment, air abrasion with Al_2O_3 particles and grinding [13]. Air abrasion with Al_2O_3 particles is generally recommended for micromechanical adhesion, but this treatment can damage the surface of the material and cause micro-cracks and loss of zirconia particles, which affect the long-term performance of the ceramic [14]. Therefore, it is assumed that surface treatment with Al_2O_3 particles may create a risk, especially for CZ with reduced strength. Moreover, this risk may increase further in restorations with minimum thickness [15].

New surface treatments have been tried in an attempt to improve adhesion properties by increasing the surface energy without affecting the properties of materials. Nonthermal argon plasma (NP) application is one such treatment, which acts by modifying surfaces [16]. As confirmed by many authors [1,17,18], this method increases the adhesion between resin cement and zirconia without any deleterious effects on the structure of the material, thereby reducing the risk of cracking. Nonthermal plasmas can be produced by gases such as oxygen, argon, nitrogen and helium at different rates to increase the surface energy and hydrophilicity of Y-TZP zirconia [19]. The interaction between plasma ions and electrons increases surface reactivity by breaking down stabilized radicals. This creates a more hydrophilic surface on materials such as ceramics [20]. Although some studies have investigated the effect of NP on bond strength [1,17], its effect on the mechanical strength and endurance of zirconia has not been examined.

Correspondence to Dr. Hande Citir Yucel, Department of Prosthodontics, Faculty of Dentistry, Alanya Alaaddin Keykubat University, Mustafa Kemal Avenue #82, Alanya/Antalya 07490, Turkey
Fax: +90-242-510-6009 E-mail: hande.yucel@alanya.edu.tr

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Table 1 Types and compositions of the zirconia materials and their manufacturers

Material (Abbreviation)	Type	Composition wt%	Manufacturer	Lot no.
Lava Plus (LP)	TZ	94% ZrO_2, 6% Y_2O_3, and 0.05% ≤ Al_2O_3	3M ESPE, St. Paul, MN, USA	D4GJF
InCoris TZI-C (IC)	TZ	92.4% ZrO_2, 5.5% Y_2O_3, and 0.04% Al_2O_3	Dentsply Sirona Dental Systems GmbH, Bensheim, Germany	2016289996
StarCeram Z-Smile (SC)	CZ	99% < $ZrO_2/HfO_2/Y_2O_3$, 8.50-9.60% Y_2O_3	H.C.Starck Ceramics GmbH, Selb, Germany	50606662
Katana UTML (KU)	CZ	87-92% ZrO_2, 9.3-11% Y_2O_3, and other <2%	Kuraray Noritake Dental Inc., Miyoshi, Japan	DNXJR

TZ, tetragonal zirconia; CZ, cubic zirconia

To increase the clinical longevity of restorations, it is important to ensure that any surface treatment has only minimal damage to CZ and TZ monolithic zirconia materials. Furthermore, literature regarding the fracture resistance of new CZ restorations after different surface treatments is limited.

The purpose of the present study was to evaluate the effect of air abrasion with Al_2O_3 and NP surface treatment on the fracture resistance of TZ and CZ restorations. The null hypotheses were that (i) the surface treatment type and (ii) the zirconia material used would have no influence on the fracture resistance of zirconia restorations.

Materials and Methods

Four types of zirconia material (two tetragonal and two cubic) and two different surface treatment methods (air abrasion with 110 µm Al_2O_3 and NP) were used. The zirconia materials and their properties are shown in Table 1. In total, 80 monolithic zirconia restorations were produced by computer-aided design and computer-aided manufacturing (CAD-CAM) technology. Twenty restorations made of each zirconia material were further divided into two subgroups (n = 10) for each material according to the surface treatment employed: air-abraded with 110 µm Al_2O_3 or NP.

Preparation of metal dies and zirconia restorations

Stainless steel dies were used as abutments for fracture resistance testing [21]. A 3D model was computer-designed to simulate a mandibular first molar tooth preparation (SketchUP Design Software, Trimble Inc., Sunnyvale, CA, USA), and the preparation principles were considered. The preparation involved a flat-topped metal die with an occluso-gingival height of 4 mm, a shoulder margin width of 1 mm, and a convergence angle of 6 degrees. The metal die produced on the basis of the aforementioned model (Spinner, 4-axis machine, Sauerlach, Germany) is shown in Fig. 1a. After confirming that the first metal die had achieved the desired properties, 80 metal dies were prepared using the same technique.

The metal die preparations were scanned using a 3D digital scanner (3Shape Trios, Copenhagen, Denmark). The TZ and CZ restorations were also prepared with a CAD-CAM system (HinriMill 5, Goslar, Germany) from pre-sintered zirconia disks. All restorations had an occlusal thickness of 0.5 mm, a wall thickness of 0.8 mm [22], and an intaglio clearance of 80 µm [23]. The restorations were prepared in an occlusal coping style without cusps and central fossa to ensure the same standard occlusal morphology, as shown in Fig. 1b. The restorations were subjected to final sintering in accordance with the manufacturer's instructions.

Surface treatments

Before surface treatments the restorations were cleaned with distilled water in an ultrasonic cleaner for 10 min and dried. The inner surfaces of the air abrasion groups were air abraded with 110 µm Al_2O_3 particles (Korox, Bego, Bremen, Germany) from a distance of 10 mm and under a pressure of 0.4 MPa for 15 s. For the NP treatment, a nonthermal atmospheric pressure plasma (NTAPP) jet generating device was used. The NTAPP jet assembly consisted of a 150 mm-long quartz glass tube with outer and inner diameters of 6 mm and 4 mm, respectively. Argon was fed through the glass tube at a fixed flow rate of 5 standard liters per minute. A home-made power supply was developed to generate the plasma using a sinusoidal high voltage. The tip of the plasma source was positioned vertically 10 mm above the inner surface of the zirconia specimen to allow application of the nonthermal plasma. The duration of plasma exposure was 2 min for each sample.

Scanning electron microscopy (SEM) observation

One extra sample from each subgroup was prepared to examine the topography of the treated surfaces. After the treatment procedure, the surfaces of the samples were examined using a SEM at ×1,000 magnification (Zeiss EVO LS 10, Oberkochen, Germany). After surface treatments, air-abraded restorations were ultrasonically cleaned in an ethanol solution for 10 min and dried. Ultrasonic cleaning was not applied to the restorations with NP surface treatment in order not to damage the active surfaces. The changes on the surface of the zirconia were then interpreted.

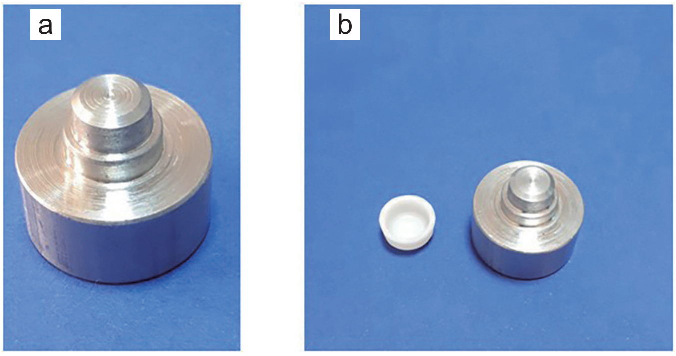

Fig. 1 Photographs of a) the stainless steel die and b) the zirconia restoration used in this study

Cementing and thermocycling

All restorations were cemented to the metal dies with a self-adhesive resin cement (Panavia SA Plus, Kuraray Noritake Dental Inc., Miyoshi, Japan) at room temperature (25°C). The restorations were cemented with finger pressure and subjected to a constant static load of 7 kg for 5 min during polymerization. After polymerization, the samples were kept in distilled water for 24 h at 37°C, and then thermal cycling was applied to all groups for aging. Thermal cycling was carried out at 10,000 cycles in water at 5°C to 55°C, with a 30-s dwell time at each temperature. Thereafter, the fracture resistance values of the restorations were measured.

Measurement of fracture resistance

Fracture resistance testing was performed with a universal testing device (Lloyd instruments, Beijing, P. R. China). During the application of force, the sample was fixed to the table of the device so that it remained motionless at a right angle. The crosshead speed was set at 0.5 mm/min. Compressive force was applied vertically towards the midpoint of the occlusal surface of the specimen with a 3-mm-diameter steel ball. Force was increased until the first fracture occurred [21]. The forced being applied at the moment of fracture was recorded in the software of the device in Newtons (N).

Statistical analysis

Before the data were subjected to statistical analysis, continuous variables were examined with the Shapiro-Wilk test (P = 0.886) for normality and Levene's test (P = 0.367) for homogeneity of variance. Since the parametric test assumptions were confirmed, intergroup evaluation for the two categorical variables was performed using two-way analysis of variance (ANOVA). Th significance of differences resulting from this analysis was determined by the Bonferroni multiple comparison test. The SPSS 14.01 software package was used for analysis of the data (IBM SPSS Statistics, Armonk, NY, USA). Differences at $P < 0.05$ were considered to be significant.

Results

Two-way ANOVA revealed statistically significant differences in mean fracture resistance among the four zirconia materials and the two surface

Table 2 Results of two-way ANOVA of the mean fracture resistance values (N)

Effect	Type III sum of squares	df	Mean square	*F*	Sig
Zirconia material	3094081.338	3	1031360.446	10.787	.000
Surface treatment	3215619.013	1	3215619.013	33.633	.000
Zirconia material × surface treatment	1243081.237	3	414360.412	4.334	.007

*Significantly different at $P < 0.05$

Table 3 The mean and standard deviations (SD) of the mean fracture resistance values (N) for the test groups

	Surface treatment	
Material	Air abrasion	Nonthermal argon plasma (NP)
KU	1,446.1 ± 415.5^{Aa}	1,376.1 ± 345.4^{Aa}
SC	2,114.0 ± 369.8^{Bb}	1,789.9 ± 142.4^{Cb}
IC	2,167.8 ± 266.4^{Bb}	1,404.8 ± 257.3^{Ca}
LP	1,892.9 ± 362.8^{Bb}	1,454.3 ± 218.6^{Ca}

Means followed by different superscript letters differ significantly at the 0.05 confidence level. Upper case: significant differences between rows; lower case: significant differences between columns

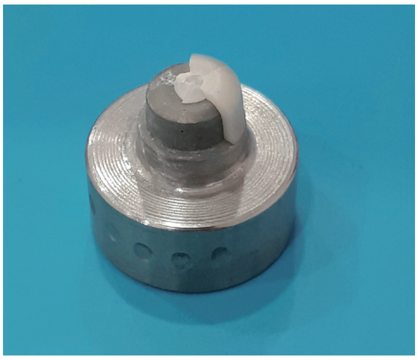

Fig. 2 Fracture pattern of zirconia restorations

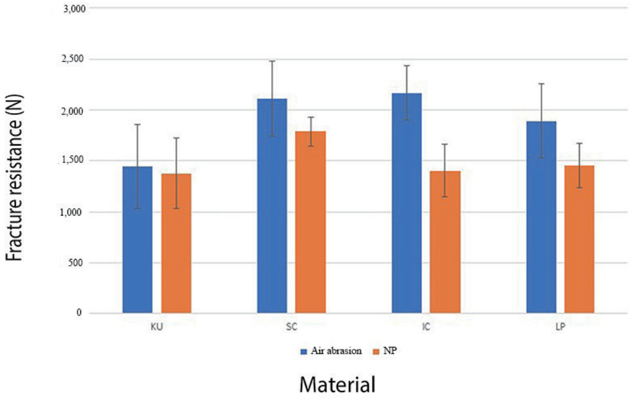

Fig. 3 Fracture resistance (N) of zirconia materials according to surface treatment

treatments ($P < 0.001$). Their interaction was significant for fracture resistance ($P < 0.05$) (Table 2). Mean fracture resistance values (N) and standard deviations for the study groups are presented with the Bonferroni *post hoc* comparison results in Table 3.

All of the TZ and CZ restorations demonstrated similar fracture patterns after resistance testing. Fractures that initiated under the loading point and propagated through the inner surface of the restorations were irreparable (Fig. 2).

The highest mean fracture resistance value (2,167.8 ± 266.4 N) was observed for the InCoris TZI-C (IC) zirconia restorations in the air-abraded group while the lowest value was observed for the Katana UTML (KU) zirconia restorations in the NP-treated group (1,376.1 ± 345.4 N) (Fig. 3). For all zirconia materials, while the restorations in the air-abraded group showed higher mean fracture resistance values than those in the NP-treated group, significant differences were observed among the StarCeram Z-Smile (SC) ($P = 0.019$), IC ($P < 0.001$) and Lava Plus (LP) ($P = 0.004$) restorations. KU restorations showed no significant difference in mean fracture resistance ($P = 0.687$) between the two surface treatments.

In the air-abraded groups, there were significant differences in mean fracture resistance among the SC/KU ($P = 0.001$), IC/KU ($P < 0.001$) and LP/KU ($P = 0.044$) zirconia restorations. There were no significant differences among the SC/LP, SC/IC ($P = 1.000$) and LP/IC ($P = 0.624$) zirconia restorations. IC zirconia restorations had the highest mean fracture resistance, whereas KU zirconia restorations had the lowest.

In the NP-treated groups, there was a significant difference between the mean fracture resistance values for the SC/KU ($P = 0.005$), SC/IC ($P = 0.009$) and SC/LP ($P = 0.031$) zirconia restorations, but no significant differences were evident among the KU/IC, KU/LP and IC/LP ($P = 1.000$) zirconia restorations. The SC group had the highest mean fracture resistance, whereas the KU group had the lowest.

SEM analysis results

SEM images at ×1,000 magnification after the two different surface treatments are shown in Fig. 4. After NP surface treatment, no deformation or roughness was apparent. Irregular surfaces and roughness were observed in all of the air-abraded zirconia materials.

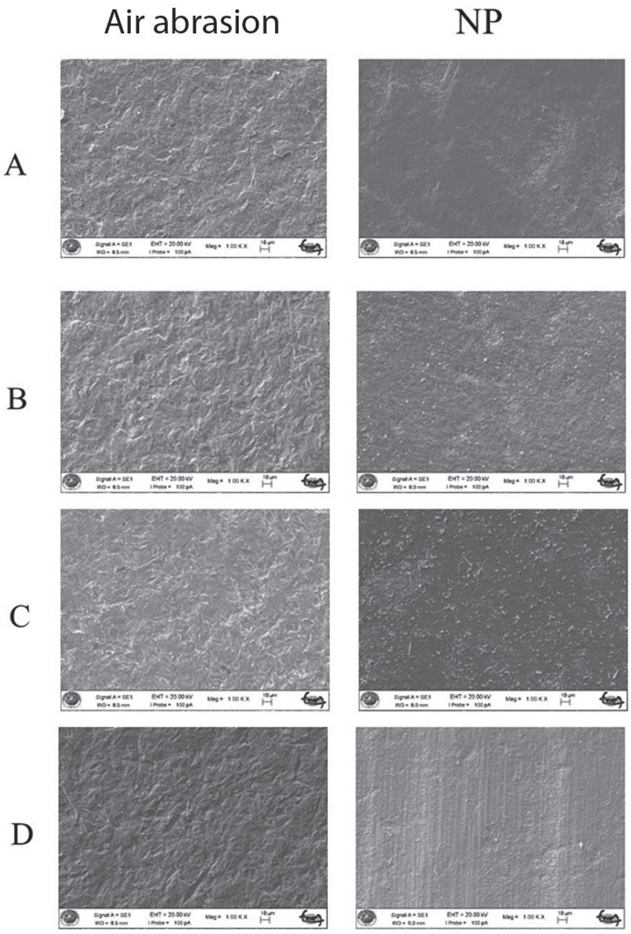

Fig. 4 SEM images of the materials used in this study after surface treatments. (A) Katana UTML zirconia, (B) InCoris TZI-C zirconia, (C) StarCeram Z-Smile zirconia, and (D) Lava Plus zirconia (×1,000 magnification)

Discussion

In the present study, the fracture resistance of TZ and CZ restorations after two different surface treatments was evaluated. It was found that the type of surface treatment and zirconia material significantly affected the fracture resistance. Therefore, the null hypotheses of the study were rejected.

The mean fracture resistance values of air-abraded zirconia restorations ranged between 1,446.1 ± 415.5 N and 2,167.8 ± 266.4 N, whereas those of NP-treated zirconia restorations ranged between 1,376.1 ± 345.4 N and 1,789.9 ± 142.4 N. All of the air-abraded restorations showed higher fracture resistance than the NP-treated restorations. Air abrasion improves micromechanical retention by roughening the zirconia surface, allowing the resin cement to penetrate the defects and rough contours. This increase of surface area can improve the mechanical behavior of zirconia [24]. Fernandes et al. [1] found that NP did not significantly alter the surface roughness of zirconia materials. Likewise, in the present study, NP surface treatment did not create rough areas like those resulting from air abrasion, as seen in the SEM images. In addition, the air abrasion procedure generates compressive stress on the surface of zirconia, causing t-m phase transformation and resulting volumetric expansion that prevents crack propagation and increases the durability of the material [25]. NP surface treatment is a cold procedure that does not generate heat in the material [1]. Therefore, t-m phase transformation is less likely to occur. Furthermore, the phase transformability of zirconia depends on its microstructure, the amount of yttrium in the material, and hence the cubic phase content [26]. Cubic crystal structure in zirconia does not cause phase transformation under stresses such as air abrasion, grinding and wetting. An increase in the tetragonal crystal structure promotes phase transformability [10,27]. In the present study, there was no significant difference of mean fracture resistance between the air-abraded and NP-treated KU (cubic zirconia) groups. However, the mean fracture resistance in the air-abraded group was slightly higher than that in the NP-treated group. This may have been attributable to the roughness created by air abrasion on the surface of the material with low phase transformation ability.

Lawson et al. [22] investigated the effect of cement and surface treatment on the fracture load of traditional zirconia, translucent zirconia, and lithium disilicate restorations. They found that 3Y-TZP tetragonal zirconia restorations adhering to resin cements had a higher fracture load than 5Y-TZP translucent zirconia restorations ($P < 0.0001$). However, no significant difference was found between 5Y-TZP and lithium disilicate restorations. Kwon et al. [28] compared flexural strength among 5Y-TZP, 3Y-TZP, and lithium disilicate zirconia bars. They found that 5Y-TZP zirconia bars had lower flexural strength than 3Y-TZP zirconia bars. Elsayed et al. [29] also examined the fracture strength of 3Y-TZP, 4Y-TZP and 5Y-TZP monolithic zirconia restorations and found that the increase in the yttrium content had a deleterious effect on the mechanical properties. Although these previous studies indicated that TZ had higher fracture resistance than CZ, the present study demonstrated differences in fracture resistance between two different surface treatments. In the air-abraded groups, tetragonal zirconia restorations (IC and LP) showed higher mean fracture resistance values than KU cubic zirconia restorations. However, mean fracture resistance of air-abraded SC cubic zirconia restorations was similar to that of air-abraded IC and LP tetragonal zirconia restorations. This may have been because the tetragonal structure within cubic zirconia affects the mechanical properties, as previously stated by Zhang et al. [26]. In their study, two 5 mol% zirconia differing in tetragonal grain structure and microstructure but having a similar phase composition (60% c- and 40% t- ZrO_2) exhibited significant differences in mechanical properties and aging stabilization. They considered that besides the high content of cubic grains in the zirconia, the crystal properties and microstructure of the tetragonal grains had also affected its properties.

Inokoshi et al. [27] found that air abrasion reduced the durability of zirconia material with a high yttrium content when microcracks occurred. They confirmed that after air abrasion, the flexural strength of Katana HT (4 mol%) – which had the lowest yttrium content – increased the most, followed by Katana STML (5 mol%) and Zpex Smile (5 mol%), whereas the flexural strength of Katana UTML (6 mol%) decreased. Their results suggested that the flexural strength of the latest-generation zirconia materials after air abrasion was directly related to the composition and microstructure of the zirconia. This is because the durability of zirconia after air abrasion is determined by the balance between the presence of microcracks and the generation of surface compressive stress. Therefore, it can be concluded that processes that do not cause cracks on the surface, such as NP, may be more suitable for the surface treatment of zirconia with low t-m phase transformation ability. Future studies are needed to clarify the most appropriate surface treatment for cubic zirconia with a higher yttrium content.

On the other hand, it is known that the durability of zirconia decreases as a result of continuous wetting with saliva, sudden changes in temperature, and lateral forces [30]. Zirconia is susceptible to LTD, which may occur in the oral environment when surfaces are in contact with water within the oral temperature range [2,31]. Thermal cycling is a procedure that simulates oral conditions and is better for determining the LTD of zirconia [32]. In this study, thermal cycling was applied to all study groups to simulate conditions in the oral environment.

LTD may also be another reason for the significantly lower fracture resistance values of the NP-treated tetragonal (IC and LP) and cubic SC zirconia restorations. Kengtanyakich and Peampring [33] investigated the mechanical properties and hydrothermal degradation of disk-shaped 3Y-TZP and cubic zirconia materials. They found that cubic zirconia had lower fracture hardness and flexural strength than 3Y-TZP before aging. After aging, however, while a significant decrease of durability was observed for 3Y-TZP, there was no such decrease for zirconia with a high amount of cubic phase. Air abrasion, which increases the durability of the material by stimulating t-m phase transformation, may have increased the resistance to LTD [34,35]. On the other hand, NP surface treatment, which does not have such an effect, may have reduced the durability of zirconia with high amount of tetragonal grains. The results of the present study suggest that air abrasion is a more advantageous surface treatment than NP for zirconia materials with a low resistance to LTD. Therefore, further studies are necessary to investigate the effects of air abrasion and NP surface treatments on the LTD of tetragonal and cubic zirconia.

The conditions of air abrasion with Al_2O_3 (pressure, distance, particle size, time) affect the strength of zirconia material. Okada et al. [36] reported that the surface roughness of zirconia increased when air abrasion pressure was higher, and that micro-cracks were observed in the surface when a pressure of 0.4 MPa was employed. The same authors also indicated that air abrasion distance did not significantly change the strength of the zirconia material. A particle size of 30-120 µm is generally used for air abrasion with Al_2O_3. Larger-size particles cause more surface defects and microcracks [34,35]. Surface loss of zirconia material increases with longer air abrasion time [37]. Hence, for the present study, a 110-µm particle size, an air abrasion pressure of 0.4 MPa, a distance of 10 mm, and a time of 15 s were selected, and these were thought to create more defects on the surface. Additionally, as applied in this study, NP surface treatment from a distance of 10 mm for 2 min is considered sufficient for effective adhesion between the resin cement and zirconia material [1,16].

The mechanical strength of zirconia restorations, especially those made of monolithic zirconia, depends on the thickness of the material. Many studies have evaluated the thickness of monolithic zirconia restorations for clinically acceptable applications [13,38]. The occlusal thickness of monolithic zirconia restorations is the main factor that affects their stress and fracture strength. As reported in earlier studies, monolithic zirconia restorations with an occlusal thickness of 0.5 mm showed sufficient fracture strength against occlusal forces in the posterior region and could be used safely [6,39]. However, due to the effects of surface treatment on the durability of monolithic zirconia, this factor should be taken into account for minimum-thickness restorations [40]. Accordingly, fracture resistance testing of monolithic zirconia restorations in the present study was performed on molar restorations with an occlusal thickness of 0.5 mm.

In this study, stainless steel metal dies were used for testing the fracture resistance of zirconia restorations. Such dies have been found to be appropriate in terms of their high elastic modulus, and standardization of the die material and their preparation. For optimal fracture resistance, zirconia restorations need to be fixed without any damage to the supporting abutment. Many researchers have used metal dies to test the fracture resistance of restorations [21,39].

Differences in the sample sizes, shapes, test methods and microstructures of the materials may have affected the results of the present and previous studies. This study had certain limitations. Firstly, although thermal aging was performed, mechanical aging, which mimics masticatory forces, was not investigated. Second, the thermal cycling applied was equivalent to about 1 year in the oral cavity. These conditions should be taken into consideration in further studies.

In conclusion, this study has shown that all air-abraded TZ and CZ restorations had higher fracture resistance values than restorations subjected to NP treatment. In the NP-treated groups, SC restorations showed significantly higher mean fracture resistance than IC, LP and KU zirconia restorations. Also, in the NP-treated groups, no significant differences in mean fracture resistance were evident among the KU, IC and LP restorations. Mechanical strength is an important factor for the long-term success of zirconia restorations. Surface treatments for adhesive cementation also improve the fracture resistance. Although NP surface treatment is claimed to provide strong adhesion between zirconia material and resin cement, further research is needed to clarify the long-term effects of NP surface treatment on the durability of zirconia restorations with different microstructures.

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Conflict of interest

None of the authors have any financial interest in the companies whose materials were used in this study.

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