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Original Article

Effect of nonthermal argon plasma treatment on the surface properties and phase transformation of zirconia

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Abstract

Purpose: This study aims to evaluate the effect of applying different parameters of nonthermal argon plasma (NTAP) on the surface roughness and phase transformation of yttrium-stabilized tetragonal zirconia polycrystalline (Y-TZP) ceramics.
Methods: A total of 60 zirconia samples were prepared and randomly divided into six groups according to their surface treatments (n = 10). Group 1: control group; Group 2: argon plasma with a flow rate of 5 lt/min for 4 min; Group 3: 8 lt/min for 4 min; Group 4: 8 lt/min for 2 min; Group 5: 5 l/min for 2 min; Group 6: air abrasion with Al_2O_3 particle. The surface roughness was measured with a profilometer, and surface topography was observed using scanning electron microscopy (SEM). X-ray diffraction (XRD) analysis was performed to investigate the phase transformation.
Results: The air abrasion group showed the highest surface roughness. The lowest relative monoclinic phase amount (Xm) was observed in the control group (0.4%), and the highest Xm value was observed in group 6 (7.8%).
Conclusion: While the air abrasion group showed the highest average surface roughness, it also caused the highest phase transformation. With a flow rate of 8 lt/min for 2 min NTAP treatment increased the surface roughness without causing significant phase transformation.

Keywords: nonthermal argon plasma, phase transformation, surface roughness, Y-TZP zirconia

Introduction

In recent years, along with developments in dental applications based on computer-aided design/computer-aided manufacturing (CAD-CAM), the use of zirconia-containing ceramics has gradually increased in dental prosthetic clinics because of its superior properties such as esthetic, chemical stability, biocompatibility, and good compressive and flexural strength [1].

Zirconia is an allotropic metastable material that can be present in different crystallographic structures without deteriorating its chemical composition. Zirconia has three crystallographic phases at room temperature. Pure zirconia exists as a monoclinic crystal structure, but it transforms into a tetragonal structure at 1,170°C and a cubic structure at 2,370°C. When zirconia cools down, a tetragonal-monoclinic transformation occurs. This change in microstructure generates localized compressive stresses that are associated with a volume expansion of about 3−5%; therefore, potential crack propagation can be prevented. This mechanism is termed transformation toughening, and it is closely related to the mechanical properties of zirconia. The addition of stabilizing oxides, typically yttria (Y_2O_3), can create a tough multi-phase of partially or fully stabilized zirconia materials [2,3]. Yttrium-stabilized tetragonal zirconia polycrystalline (Y-TZP) is the most widely used ceramic for prosthetic restorations.

Despite the superior mechanical properties of Y-TZP, bonding it to other substrates is difficult because of its hydrophobic surface. Several studies have shown that untreated surfaces display low bond strength, which eventually leads to adhesive failure. Therefore, the surface properties of these ceramics need to be modified for bonding to be effective. Conventional adhesion methods (e.g., etching with hydrofluoric acid) of roughening silica-containing ceramics do not apply to zirconia because they fail to achieve adequate surface roughness, leading to poor bonding strength [4,5]. To overcome this limitation, different protocols, such as airborne-particle abrasion, tribochemical silica coating, lasers, plasma spray, using functional silane monomers, using phosphate acidic monomers, silica nanoparticle surface deposition, and a combination of protocols have been proposed to achieve reliable and long-lasting interaction between Y-TZP ceramics and resin cement [1,6,7].

Air abrasion with alumina particles is considered the gold standard surface treatment because it increases the mechanical bonding strength with porcelain veneer roughness. However, it causes micro-cracks and phase transition, which is a disadvantage in the long term [4]. Irradiation with lasers might be a potential alternative to creating a rough ceramic surface and micromechanical retention. The thermomechanical ablation caused by irradiation with an erbium-doped yttrium aluminum garnet (Er: YAG) laser can remove particles by evaporation and micro-explosions. However, conducting an irradiation protocol inappropriately may result in surface damage such as melting or cracks [8,9].

As an alternative, nonthermal plasma (NTP) activation can be considered a promising option to further improve the bond strength between zirconia and a composite in addition to or instead of mechanical surface treatment. Nonthermal gas plasma technology is based on a partially ionized gas containing highly reactive particles, such as electronically excited atoms, ions, electrons, molecules, and free radicals at near room temperature. They can react with the material's surface and modify it physically and/or chemically, improving surface energy and wettability, without affecting its properties. Studies have shown that argon plasma significantly increases the surface energy of Y-TZP dental ceramics [6,10,11]. Plasma activation resulted in a significantly smaller contact angle of adhesive on the ceramic surface, which explains the increase in surface hydrophilicity [12].

The plasma performance of these applications significantly depends on the experimental parameters, and one of the most important characteristics is the gas species. Conventional plasma sources are limited in their ability to generate gas; therefore, previous studies that experimented with the surface treatment of zirconia only used a few specific gases, including nitrogen, helium, and argon [13,14]. Additionally, the effect of irradiation time and the flow rate of plasma on the zirconia surface remains unclear [15]. None of the studies in the literature compare plasma application parameters to the surface of dental ceramics.

This study aims to apply nonthermal argon plasma (NTAP) at different flow rates and different times onto a zirconia surface to evaluate the effect on its surface roughness and phase transformation. The null hypothesis of the present study was that NTAP applied for a longer time and with a higher flow rate will not affect the surface roughness and phase transformation of zirconia.

Materials and Methods

Sample preparation

Sixty pre-sintered zirconia specimens (Kuraray Noritake Dental Inc.,

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Nagoya, Japan), 6.5 mm in diameter and 3.5 mm in height, were prepared using a CAD-CAM milling device (Sirona inLab MC XL, Bensheim, Germany). The zirconia samples were sintered for 8 h at 1,500°C in the Zyrcomat furnace (VITA Zyrcomat 6000 MS, Vita Zahnfabrik, Bad Säckingen, Germany) according to the manufacturer's instructions. A grinding machine (Phoenix Beta Grinder/Polisher, Buehler, Esslingen am Neckar, Germany) was used with 600-, 800-, and 1,200-grit silicon carbide abrasives (English Abrasives & Chemicals Ltd., Stafford, UK) for 15 s to form a standard bonding surface on the zirconia specimens.

The Y-TZP discs were randomly distributed in six groups according to the following surface treatments (Table 1):

Group 1 (control group): No surface treatment was applied to the samples.

Group 2 (plasma 4 min, 5 lt/min): Plasma was applied to the samples in this group for 4 min at a constant flow rate of 5 lt/min. Argon was applied with the tip of the plasma source positioned 10 mm above the surface of the zirconia sample.

Group 3 (plasma 4 min, 8 lt/min): Plasma was applied to the samples in this group for 4 min at a constant flow rate of 8 lt/min. Argon was applied with the tip of the plasma source positioned 10 mm above the surface of the zirconia sample.

Group 4 (plasma 2 min, 8 lt/min): Plasma was applied to the samples in this group for 2 min at a constant flow rate of 8 lt/min. Argon was applied with the tip of the plasma source positioned 10 mm above the surface of the zirconia sample.

Group 5 (plasma 2 min, 5 lt/min): Plasma was applied to the samples in this group for 2 min at a constant flow rate of 5 lt/min. Argon was applied with the tip of the plasma source positioned 10 mm above the surface of the zirconia sample.

Group 6 (air abrasion group): 110 µm Al_2O_3 powder was applied to the samples from a distance of 10 mm under 2.8 MPa pressure for 10 s with a sandblasting device (Renfert Basic Eco, Renfert GmbH, Hilzingen, Germany). The samples were then dried with oil-free air.

Nonthermal atmospheric pressure plasma jet application

The NTP process was performed in the Department of Physics at Eskişehir Osmangazi University using the nonthermal atmospheric pressure plasma jet (NTAPPJ) generating system shown in Fig. 1. The system consists of a power source, a flowmeter that measures the gas flow rate from the nitrogen gas tank, and a plasma jet system.

The plasma source consists of two concentric electrodes through which gas flows. The gas discharge is ignited by applying kHz power to the inner electrode, and the ionized gas from the plasma jet exits through a glass tube. The NTAPPJ assembly consists of a 150 mm-long quartz glass tube with an outer diameter of 6 mm and an inner diameter of 4 mm. A coaxial dielectric barrier configuration was used for the electrode geometry. A 0.5 mm-thick sharp-tipped tungsten wire was used as the power electrode and placed in a glass tube with a 4 mm inner diameter. A 5 mm-wide metal crocodile clip was used as a ground electrode and was attached to the glass tube 40 mm from the end where the discharge exits to the atmosphere (Fig. 2).

A constant argon flow rate of 8 lt/min for Groups 3 and 4, and 5 lt/min for Groups 2 and 5 was used. The peak-to-peak voltage of the 15 kHz sinusoidal waveform was optimized and then fixed at 18 kV for all experiments. A 20 mm-long plasma stream was run at room temperature (25°C). The tip of the plasma source was positioned 10 mm above the surface of the zirconia sample and placed vertically so the plasma could be applied to the entire surface. The plasma exposure time was 4 min for each sample in Groups 2 and 3, and 2 min for each sample in Groups 4 and 5.

Surface roughness measurement

The surface roughness (Ra [µm]) of the specimens was measured using a contact surface profilometer (Surftest SJ 201, Mitutoyo, Tokyo, Japan) with a 0.25 mm cutoff value. The resolution of the profilometer was 0.01 mm, the transverse length was 4.0 mm, and the diameter of the diamond recording pin stylus was 5 µm. The constant measuring speed was 0.5 mm/sec to determine the average roughness profile. Three measurements were taken at equal distances on each specimen. Ra values were recorded by taking the average of the measurements made.

Table 1 Experimental parameters

Groups	Surface treatment	*n*
1	control	10
2	plasma 4 min, 5 lt/min	10
3	plasma 4 min, 8 lt/min	10
4	plasma 2 min, 8 lt/min	10
5	plasma 2 min, 5 lt/min	10
6	air abrasion	10

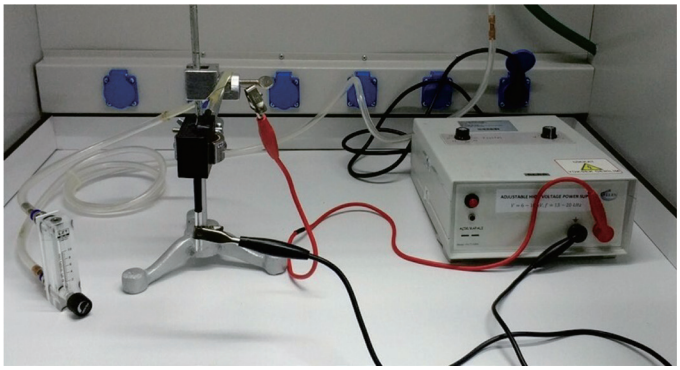


Fig. 1 Nonthermal atmospheric pressure plasma jet generating system

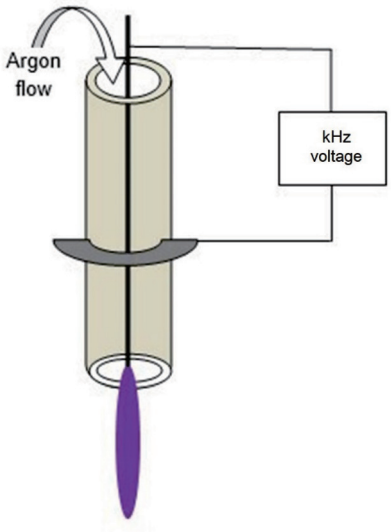


Fig. 2 Schematic representation of NTAP

SEM observation

The surface changes of the specimens were evaluated using an SEM (Zeiss Leo 1430, Carl Zeiss, Oberkochen, Germany) at 1,000 and 2,000 magnification after covering the surface with a thin layer of gold. Simultaneously, the same specimen used for SEM analysis was submitted to energy dispersive spectroscopy (EDS) at 30 kV of accelerated voltage.

Phase analysis with XRD

After the surface treatments on the zirconia samples, X-ray diffraction (XRD) analysis was performed to examine the t→m phase transformation values. Six specimens were selected randomly for XRD analysis, and their crystal structures and t/m phase transformations were examined using a diffractometer (Rigaku SmartLab, Rigaku Corp., Tokyo, Japan) with monochromatic Cu-Ka radiation (Cu-Ka l = 0.154 nm, 45 kV, 40 mA). The surface was scanned at angles between 10 and 90 degrees in 0.01-degree increments. Intensity values were recorded from the analysis results, and the 2q angles of these values were determined from the maximum values observed. The relative monoclinic phase fraction on the treated surfaces of the specimens, Xm, was calculated. The monoclinic phase fraction was expressed as the percentage of the tetragonal phase that was converted to the monoclinic phase [1].

Statistical analysis

Analyses were performed using the SPSS 23.0 (SPSS for Windows; SPSS, Inc., Chicago, IL, USA) software package with a $P < 0.05$ significance level. Assumptions of data conformity to normal distribution and homogeneity of variances were evaluated with Shapiro−Wilk and Levene tests, respectively. The surface roughness was compared between groups using one-way ANOVA. Tukey's test, one of the *post-hoc* tests, was used as a multiple comparison test to determine the group that created the difference.

Results

Surface roughness

The mean and standard deviation values of the surface roughness data are provided in Table 2.

In terms of mean surface roughness, the highest value was found in the air abrasion group (1.077 ± 0.121 µm; Group 6), and the lowest value was found in the control group (0.582 ± 0.140 µm; Group 1). The surface roughness of Group 4 was significantly higher than the control group but not significantly different from the air abrasion group. *P*-values based on Tukey's *post hoc* test results in pairwise comparisons of surface roughness are given in Table 3.

Table 2 Surface roughness and standard deviation of study samples

	n	Mean (µm)	Standard deviation
Group 1	10	0.582	0.140
Group 2	10	0.725	0.257
Group 3	10	0.870	0.261
Group 4	10	0.870	0.227
Group 5	10	0.731	0.256
Group 6	10	1.077	0.121

Table 3 *P*-values based on Tukey's *post hoc* test results in pairwise comparisons of surface roughness

Groups	Group 1	Group 2	Group 3	Group 4	Group 5	Group 6
Group 1		0.688	0.051	0.050*	0.654	<0.001*
Group 2			0.675	0.674	1.00	0.009*
Group 3				1.00	0.708	0.294
Group 4					0.707	0.295
Group 5						0.01*
Group 6						

*Significant *P*-values

Table 4 Xm values by groups

	Monoclinic phase ratio (%)
Group 1	0.4
Group 2	1.2
Group 3	5.7
Group 4	1.2
Group 5	1.0
Group 6	7.8

Phase transformation

The phase transformation values and XRD graphs of one sample from each group were obtained. The Xm values were obtained using the peak intensities from the XRD analysis and calculated as a percentage. From the XRD analysis, the tetragonal peak was observed around an angle of 300 and the monoclinic peak around an angle of 280. The Xm values of the groups are shown in Table 4. The peak values and graphs differed depending on the surface treatments (Fig. 3).

SEM analysis

The SEM images revealed that the surface irregularities increased in the samples that were treated with NTP. Deep grooves were observed in the air abrasion group (Fig. 4).

Discussion

Achieving sufficient bond strength is a major challenge in the long-term clinical success of zirconia restorations. Effective bonding requires physical and chemical processes on the ceramic surface to obtain satisfactory levels of the adhesive agent's wettability. As an alternative for modifying the chemical behavior of the ceramic surface, NTP gases capable of producing electrons, ions, and free radicals when stimulated by a glow discharge have been used because of their capacity to transform the chemical behavior of the material's surface without affecting its physical properties [11,16]. However, there is no consensus on the parameters of plasma application to achieve a more effective result. Therefore, this study was designed to examine the effect of NTAP application at different flow rates and different times on the surface roughness and phase transformation of zirconia. According to the findings, the null hypothesis that different parameters of NTAP application would not cause any difference in the examined variables was rejected.

Air particle abrasion is the most frequently used method to enhance the adhesion of Y-TZP to either ceramic or resin cement. Previous studies revealed that grit-blasting generates critical-sized flaws and defects. Additionally, grit-blasting induces the formation of monoclinic phase grains, which further impair the long-term stability of zirconia-based restorations [17,18]. In this study, both surface roughness and monoclinic phase volume fraction were influenced by surface treatment. Compared to the control group, it was observed that the surface roughness increased after the application of NTAP and air abrasion. Furthermore, the phase transformation in zirconia is a crucial tool for evaluating the surface treatment. A destructive result of surface pretreatments can be defined by the

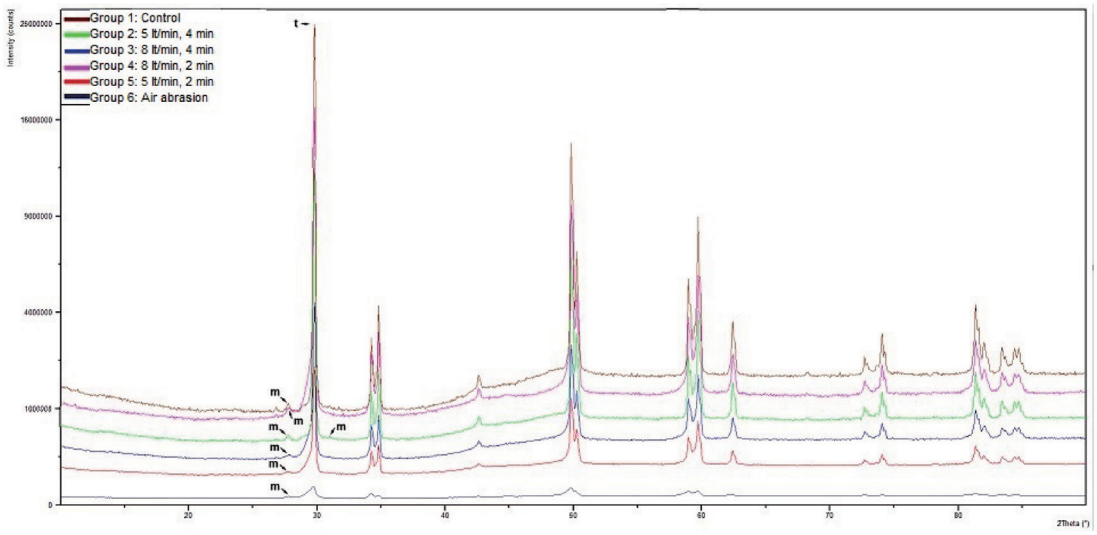


Fig. 3 Comparative view of XRD plots of samples

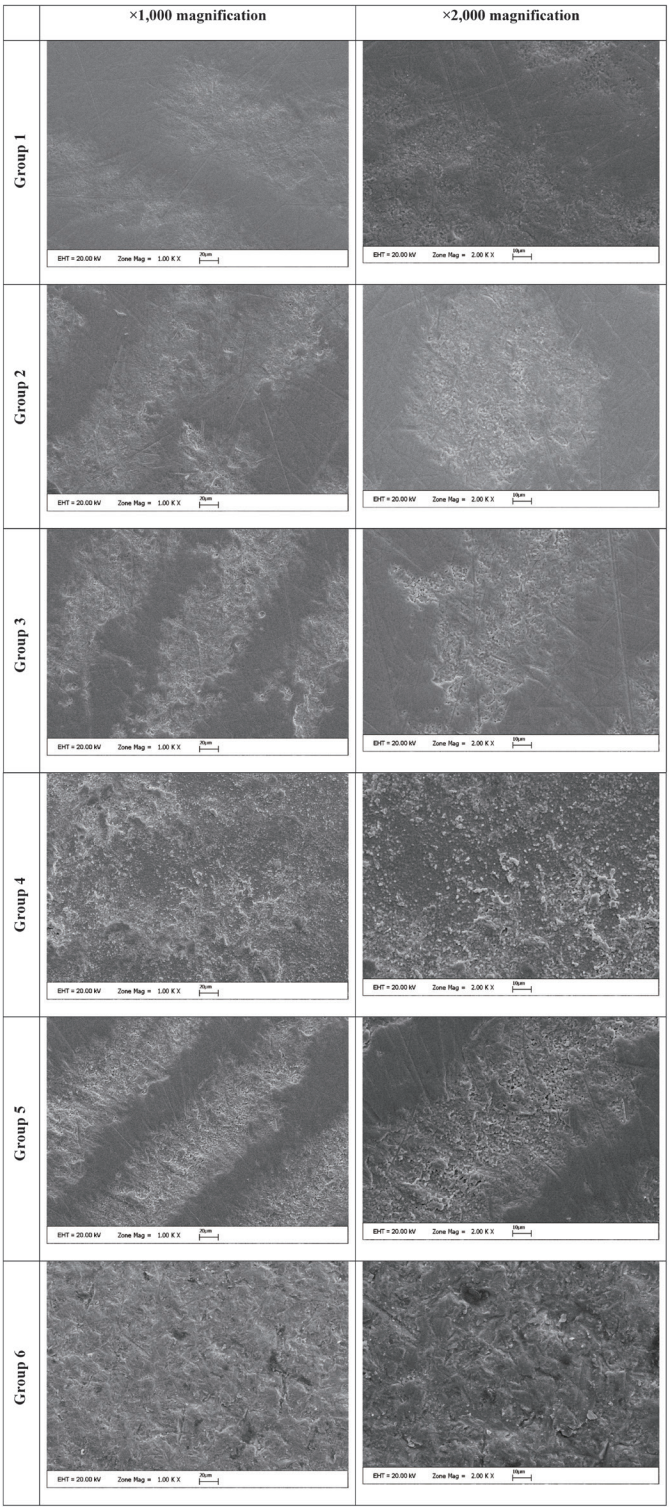


Fig. 4 Representative SEM images of Groups 1-6 after surface treatments. Group 4 and 6 showed a relatively large number of retentive and irregular areas. (×1,000 and ×2,000 magnification)

tetragonal to monoclinic phase transformation XRDs, which is a reliable method for monitoring and quantifying this transition. Surface treatment on zirconia can cause localized stress and induce its transformation from a tetragonal to a monoclinic phase, which adversely affects the mechanical properties of the material, such as its hardness, flexural strength, and modulus of elasticity. Therefore, developing a surface treatment modality that can roughen the surface with minimal phase transformation is very much desired [19,20].

The search for a surface treatment that causes fewer cracks, fractures, and tetragonal monoclinic phase transformation on the zirconia surface continues. For this purpose, Halmann et al. applied different processes on the zirconia surface and found that the ratio of the monoclinic phase was 8.6% in the group blasted with 150 μm alumina particles, 1.2% in the group blasted with 150 μm zirconia particles, and 0.68% in the applied plasma group [17]. The results of this study confirm the results of another study [21,22] that showed the ratio of the monoclinic phase on the zirconia surface is dependent on the treatment method applied to the surface. More phase transformation and surface damage were observed in the blasting groups compared to the plasma group. In this study, the group that had the highest Xm value among the plasma-applied groups was the group in which argon plasma was applied for 4 min at a flow rate of 8 lt/min. The highest phase transformation values were observed in the air abrasion group.

Ito et al. [23] compared the airborne-particle abrasion process with plasma surface treatment and reported that both surface treatments increased the shear bond strength, but there was no significant difference between them. Alumina blasting physically removes surface contaminants to improve wettability. In atmospheric-pressure low-temperature plasma treatment, C–C and C–H bonds can be broken and contaminants can be removed. XRD results on the change in crystal structure indicate that micro-cracks are generated when performing alumina blasting on zirconia, and a monoclinic structure is formed. However, although atmospheric-pressure low-temperature plasma treatment has a physical impact at the electronic level, it is believed that surface treatment occurs without modifying the zirconia crystal structure [23,24]. In this study, a significant increase in the monoclinic phase was observed in the group where NTAP was applied for 4 min at a flow rate of 8 lt/min. increasing the NTAP flow rate and application time resulted in more phase transformations.

Surface energy refers to the intermolecular forces at the surface of a material and depends on the values of the polar and dispersed components of the surface. High surface energy values indicate better surface wettability and better bonding of the material. The standard way to calculate surface energy is by measuring the contact angle of liquids [4]. In a study that examined the effects of plasma application in different combinations on a zirconia surface, it was observed that all kinds of plasma treatment (argon plasma, oxygen plasma, 20% oxygen and 80% argon plasma combination, 10% oxygen and 90% argon plasma combination) reduced the contact angle of zirconia [25]. These results agree with Noro et al. [26] and Valverde et al. [12], who attributed the reason for this phenomenon to the increase in oxygen (O) elements on the surface after the plasma application, resulting in higher polarity. It has also been reported that plasma application increases the formation of active peroxide radicals and functional groups (such as C-O and C-OH) on the treated surfaces of inert materials such as zirconia [27]. Noro et al. [26] used X-ray photoelectron spectroscopy analysis to confirm the formation of functional oxygen groups on zirconia after plasma application.

Elias et al. [7] evaluated the effect of different NTAP treatment times on the surface energy and bond strength of self-adhesive resin cement to a Y-TZP ceramic by applying NTAP to the samples for 0, 20, 60, and 120 s. They found that NTAP treatment had a positive effect on the surface energy of the samples, but there was no significant difference in terms of surface energy for different plasma treatment times. At the same time, they observed that the bond strength of the adhesive resin cement to zirconia increased as the application time of the plasma increased. This result is supported by Silva et al. who found no significant differences in the surface energy of a Y-TZP ceramic after NTAP treatment for 5, 10, or 20 s [28].

Overall, surface roughening is considered a crucial method to increase the bonding quality of resin cement to ceramic by introducing a micromechanical interlocking mechanism. Roughness parameters are useful for quantifying the microstructure of ceramic surfaces to develop structure–property relationships because quantitative microstructural analysis provides an association between the constitution, physical properties, and structural characteristics of materials [14,29]. Henningsen et al. [30] assessed and compared the changes in physicochemical surface conditions of titanium and zirconia surfaces after NTPs of argon or oxygen. There was no significant difference between the untreated and surface-treated groups concerning the surface structure, and even NTP did not change the roughness parameters or alter the microstructure. In an evaluation of similar studies in the literature that examines the effect of plasma on all-ceramic ceramics, Vechiato-Filho et al. found that plasma application did not make a significant difference to the roughness of lithium disilicate and zirconia ceramic surfaces [29]. Similarly, Çökeliler et al. reported that

no significant increase in roughness was observed in feldspathic ceramics after cold plasma application compared to the control group [31]. In this study, as per the literature, it was determined that there was no statistically significant difference in the surface roughness values of the plasma-applied groups compared to the control group. This result, in general, confirms the aggressive effect of airborne-particle abrasion on roughening the surface, which is consistent with many studies in the literature.

The limitation of this study is that the effect of NTAP application at different times and flow rates on the bond strength of zirconia with resin cement was not investigated. In conclusion, this study demonstrated that argon plasma irradiation affected the surface topography of zirconia. Furthermore, according to XRD analysis, although the highest monoclinic phase ratio was seen in the blasting group, it was determined that it did not increase in the NTAP-applied groups compared to the control group, except for Group 3. Considering the other potential drawback of alumina blasting mentioned earlier, plasma treatments could be potential approaches to modifying Y-TZP and enhancing its surface reactivity; however, more research on this subject is required.

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Conflict of interest
The authors of this manuscript certify that they have no proprietary, financial, or other personal interest of any nature or kind in any product, service, and/or company that is presented in this article.

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