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Research article

Effects of different plasma treatments on bonding properties of zirconia

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ABSTRACT

This in vitro study was to evaluate the effect of different non-thermal atmospheric pressure plasma (NTP) on shear bond strength (SBS) between yttria-stabilized tetragonal zirconia polycrystal (Y-TZP) and self-adhesive resin cement. In this study, The Y-TZP specimens were divided into 4 groups according to the surface treatment methods as follows: Control (no surface treatment), Sb (Sandblasting), AP(argon NTP), and CP(20 % oxygen and 80 % argon combination NTP). Y-TZP specimens were randomly selected from each group to observe and test the following indexes: scanning electron microscope to observe the surface morphology; atomic force microscope to detect the surface roughness; contact angle detector to detect the surface contact angle; energy spectrometer to analyze the surface elements. Then, resin cement (Rely X–U200) was bonded to human isolated teeth with Y-TZP specimens to measure SBS. The results showed that for the SE test, the NTP group was significantly higher than the control group ($p < 0.05$). The results of the SBS test showed that the SBS values of the NTP group were significantly higher than those of the other groups, regardless of the plasma treatment ($p < 0.05$). However, there was no significant difference between groups AP and CP in a test of SBS ($p > 0.05$). This study shows that non-thermal atmospheric pressure plasma can improve the shear bond strength of Y-TZP by increasing the surface energy. The addition of oxygen ratio to argon is more favorable to increase the shear bond strength and is worth further investigation.

1. Introduction

Zirconia has attracted a great deal of interest in the dental field as a restorative material. With the popularity of computers and the development of computer-aided design and computer-aided manufacturing (CAD-CAM), yttrium-stabilized tetragonal zirconia polycrystals (Y-TZP) have become the most widely used choice by dentists and patients for their excellent biocompatibility and chemical stability [1,2]. Compared to conventional zirconia, tetragonal polycrystalline zirconia with the addition of a yttrium oxide stabilizer

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substantially increases the phase transition temperature range, allowing zirconia to be maintained in a sub-stable tetragonal phase structure at ambient temperature [3]. On the one hand, transformation toughening gives zirconia high flexural and fracture strengths [4]. On the other hand, compared to metallic restorative materials, zirconia is more aesthetic and natural through the study of improved light transmission properties [5]. Zirconia veneers have become a popular choice for restoring the appearance of anterior teeth while preserving dental tissue. They offer excellent aesthetic properties [6].Y-TZP has a wide range of applications in prosthodontics due to its superior mechanical and aesthetic properties.

An important factor determining the success of dental restorations is the adhesion between the zirconia and resin cement. Y-TZP is more acid resistant than conventional silica-based ceramics and therefore does not produce a sufficiently rough surface to enhance the micromechanical forces and makes it challenging to form chemical bonds with resin cement [7]. The increase in shear bond strength (SBS) consists of two sources: physical roughening of the zirconia surface, formation with locking effect, and chemical modification [8]. Sandblasting roughs the Y-TZP surface, provides micromechanical interlocking, and is the most common physical roughening method to improve the SBS of Y-TZP and resin cements [9]. However, the microporosity produced by sandblasting treatment while changing the surface morphology of Y-TZP may trigger the development of internal cracks in Y-TZP and affect durability. This may affect the clinical application of Y-TZP restorations [10]. Therefore, a hot issue in materials science research is focused on exploring an optimal treatment to improve the shear bond strength of Y-TZP and resin cements without significant effect on mechanical properties.

Plasma is the most dominant state of matter in the universe. It is a fully or partially ionized neutral gas containing electrons, ions and neutral particles. It is considered the fourth state of matter in addition to solids, liquids and gases [11]. Compared to other methods of zirconia surface treatment, plasma has distinct advantages. It modifies only the material surface, typically by a few to a dozen nanometers, without affecting the properties of the substrate. Current applications of plasma in dental medicine include root canal disinfection and sterilization, tooth whitening, and material surface modification [12–14]. The results of Valverde [15]showed for the first time that the surface of zirconia was chemically modified after non-thermal atmospheric pressure plasma(NTP) treatment, which increased the surface energy and enhanced the SBS of Y-TZP to resin cement. NTP contains a large number of reactive groups that give it some special properties that distinguish it from ordinary gases. Previous studies have shown that NTP promotes the formation of reactive groups on the Y-TZP surface through mild etching and chemical functionalization. This resulted in a reduction of carbon content on the Y-TZP surface and enhanced surface wettability, thus improving the shear bond strength without causing significant changes in the surface morphology and mechanical properties of Y-TZP [16,17]. Furthermore, the NTP treatment process is a simple and environmentally friendly method that effectively cleans material surfaces without producing harmful pollutants. All of these features are conducive to its use in future clinical applications. However, the plasma treatment effects of different kinds of gases show different outcomes. At present, there is no uniform conclusion on the type of gas, and finding the best plasma gas is crucial.

Therefore, this study aimed to investigate the effect of NTP with different gases on the SBS between Y-TZP and resin cement. NTP combining Ar and O_2 was applied to the Y-TZP surface, and the surface energy after treatment was analyzed and the bonding strength was measured. Two null hypotheses will be tested in this study. The first hypothesis is that the different treatments will have no effect

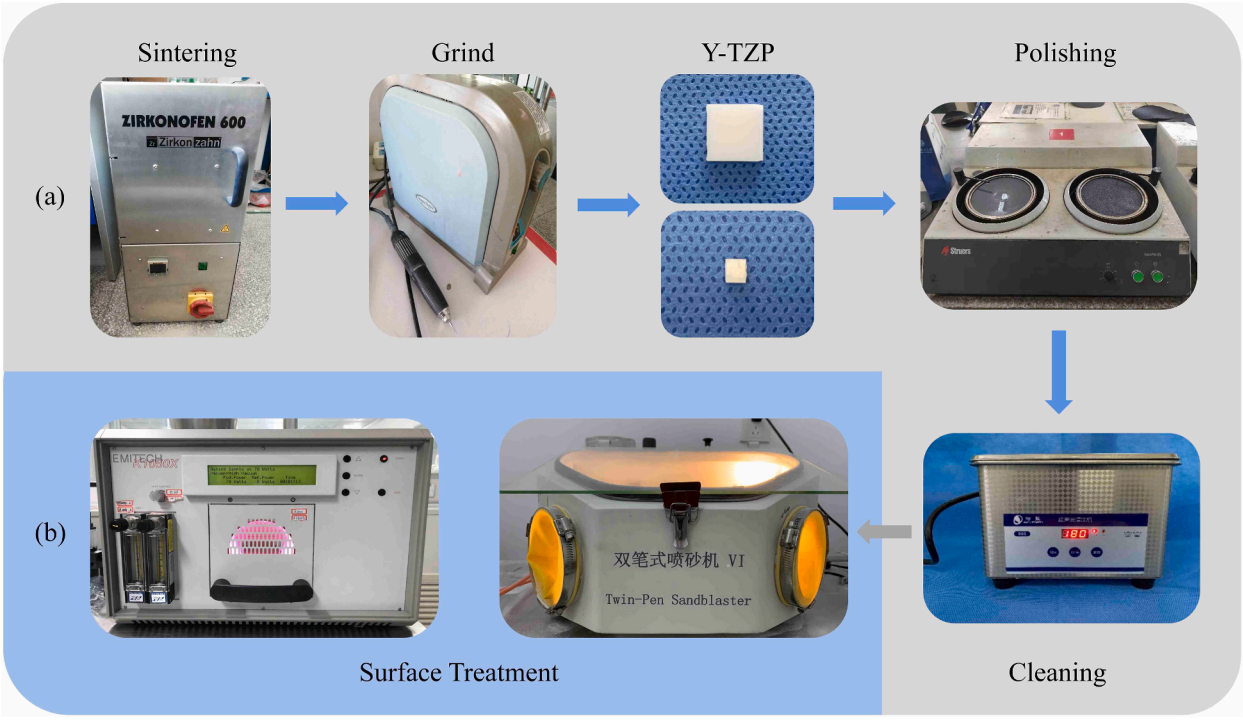


Fig. 1. Preparation process and treatment method of Y-TZP specimens.(a) Fabrication of Y-TZP specimens.(b) Surface treatment method.

on the surface energy of Y-TZP and on the SBS between Y-TZP and resin cement. The second hypothesis is that the surface energy of Y-TZP and the SBS between Y-TZP and resin cement will not be affected by NTP treatment with different gases.

2. Material and equipment

2.1. Preparation of specimens

The pre-sintered Y-TZP (Wieland Dental, Zenostar Zr Translucent T0, Germany) was sintered in a high-temperature sintering furnace (Zirkonzahn, Zironofen 600, Italy) according to the sintering procedure recommended by the manufacturer. The sintering temperature rise process is divided into 4 stages. After reaching 1550 °C, the temperature is cooled down too and stored in reserve. The Y-TZP specimens were ground and polished using a grinding machine (Marathon, Multhi600, Korea). 96 Type I Y-TZP specimens (10mm × 10mm × 2 mm) and 40 Type II Y-TZP specimens (3mm × 3mm × 2 mm) were prepared. All the Y-TZP specimens were polished using a grinding and polishing machine (Struers, Labopol-25, USA) under 600, 800, 1000, 1500, and 2000 grit silicon carbide paper. Then these specimens were placed in a numerically controlled ultrasonic cleaner (KQ-400DE, Kunshan Ultrasound Instrument Co., Ltd, China), cleaned and shaken with distilled water for 15 min, and dried for later use (Fig. 1a).

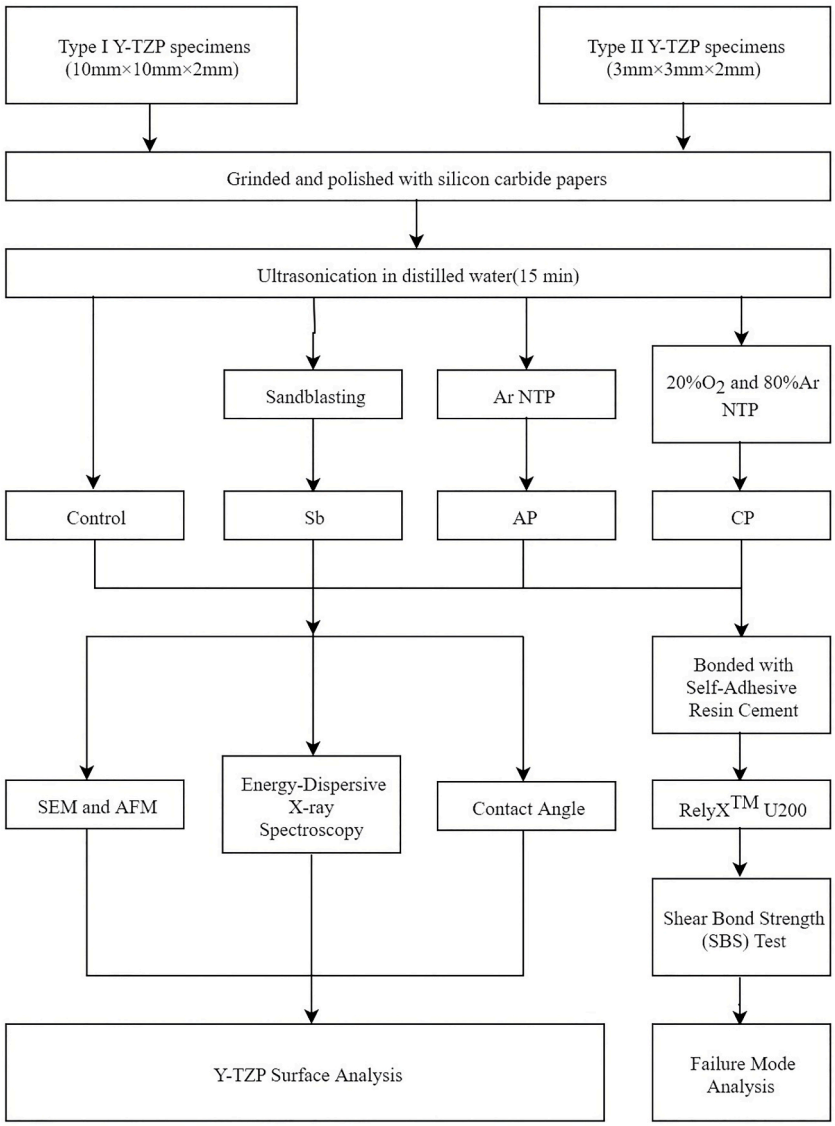


Fig. 2. Study design flow chart.

2.2. Surface treatment

Type I Y-TZP specimens were randomly divided into four groups (n = 24) and Type II Y-TZP specimens were randomly divided into four groups (n = 10) according to the following treatments:

Group Control: No surface treatment.

Group Sb: Y-TZP + Sandblasting,

Group AP: Y-TZP + argon NTP treatment.

Group CP: Y-TZP + 20 % oxygen and 80 % argon combination NTP treatment.

The Y-TZP was treated using a Twin-Pen Sandblaster (Twin-Pen Sandblaster, China). At a distance of 10 mm from the surface of Y-TZP, 110 μm Al_2O_3 particles were selected for vertical sandblasting at a pressure of 4 bar for 20s. After sandblasting, all Y-TZP specimens were cleaned by ultrasonic shock in distilled water and dried. The Y-TZP specimens were placed in the reaction chamber of the plasma generator (Quorum K1050X, UK) and fed with Ar gas and 20 % oxygen and 80 % argon combination gas respectively.At a gas flow rate of 4L/min and power of 90w for a treatment time of 120s(Fig. 1b).Fig. 2 summarizes the study design process.

2.3. Surface analysis

2.3.1. SEM and AFM

Each specimen was sputter-coated with gold and evaluated under a scanning electron microscope (SEM, ZEISS SUPRA 55, Germany) at 5000 × magnifications to assess changes in surface topography. The surface roughness of the specimens was measured using an atomic force microscope (AFM, JPK NanoWizard Ultra Speed, Germany). The wide range was 3μm × 3 μm, and each group of AFM images and roughness values were obtained using JPK data processing software. The roughness values of 6 specimens in each group were measured, and the average roughness value of the group was calculated.

2.3.2. Energy-dispersive X-ray spectroscopy

The surface elemental composition was analyzed using energy dispersive X-ray spectroscopy. The carbon and oxygen element percentages and the carbon-to-oxygen ratio were determined. The average value of 6 specimens was calculated as the result of the surface elemental composition of the group.

2.3.3. Contact angle

A contact angle goniometer (KRUSS DSA100, Germany) is used to measure the contact angle of specimens surfaces. 3 μL of distilled water is applied to the surface of the Y-TZP specimens, and the contact angle is measured after the droplet flow equilibrium. The average of each specimen's left and right contact angles was used as the measurement value. The average contact angles was calculated for each group of 6 specimens(Fig. 3).

2.4. Shear bond strength (SBS) test and failure mode

2.4.1. Bonding Y-TZP with self-adhesive resin cement

40 anterior teeth or premolars were selected for the study. The teeth were extracted from humans and were free of caries and cracks, with intact enamel on the labial side. They were cleaned using ultrasonic shock and refrigerated in saline solution.The collected human isolated teeth were truncated along the cervical part of the tooth, and the crown part was retained(Fig. 4a). Then they were put into 10mm × 20mm × 40 mm molds and filled with plaster for embedding, during the embedding process, the crowns of the isolated teeth were made higher than the plaster plane, exposing the intact labial and buccal surfaces. After the embedding was completed, the

Fig. 3. The process of measuring contact angle.

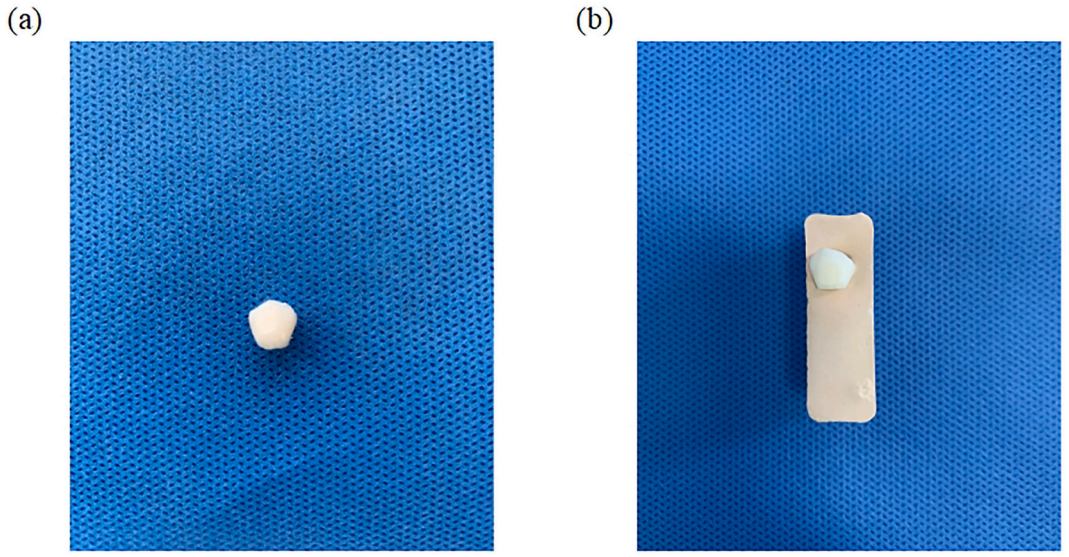


Fig. 4. (a) Crown of isolated human tooth. (b) Plaster-embedded human isolated crowns.

human isolate crowns were polished using 600, 800, 1000, 1500, and 2000 grit silicon carbide paper to prepare a plane of enamel with an area greater than 3mm × 3 mm and a thickness of less than 1 mm. Wash and dry for later use(Fig. 4b).

The differently treated Type II Y-TZP specimens were bonded directly to the prepared human isolated teeth without any zirconia primer. 3 M RelyX™ U200 self-adhesive resin cement (RelyX™ U200, 3 M, USA) was applied to the Y-TZP pre-bonded surfaces for bonding to the human isolated teeth(Fig. 5a). The specimens were fixed on a universal test machine (Instron, America) with a pressure of 10 N applied vertically for a duration of 10s(Fig. 5b). The specimens were pre-cured for 2–3s using a light-curing lamp (WOODPECKER, China). After removing the excess adhesive around the specimens, the specimens were cured for another 20s each.

2.4.2. SBS measurement

The groups of Y-TZP specimens were fixed on the fixture of the universal test machine (Instron, America) after the bonding was completed. The speed parameter was set to 0.5 mm/min to ensure that the loading head descended parallel and uniformly with the bonding interface, eventually until the Y-TZP specimens fell off the tooth surface(Fig. 6). The maximum load at the moment of fracture was recorded using a computerized microforce tester, and the shear bond strength was calculated.

2.4.3. Failure mode

The failure mode interface of each group of Y-TZP specimens after the shear experiment was completed was observed under a stereomicroscope (NSZ-606, Yongxin, China). The failure mode data of each group were recorded.

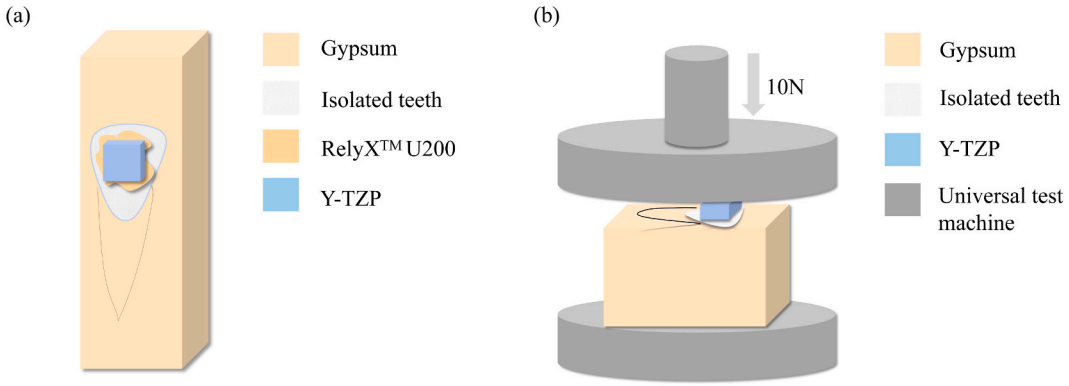


Fig. 5. Schematic diagram of preparation method for shear bond strength (SBS) Y-TZP specimens. (a) Y-TZP specimens surface and isolated teeth bonding. (b) Universal testing machine continuous pressure bonding.

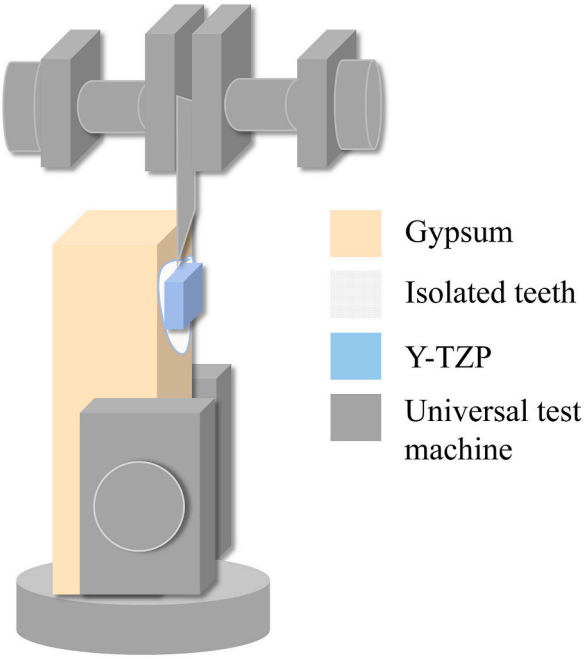


Fig. 6. Schematic figure of shear bond strength (SBS) test.

2.5. Statistical analysis

The obtained data were analyzed using SPSS statistical software(IBM SPSS Statistics 26, USA). After the regular distribution test and chi-square test, the data were subjected to a one-way analysis of variance (ANOVA) combined with the least significant difference (LSD) test, and $p < 0.05$ was considered statistically significant.

3. Results

3.1. Surface analysis

3.1.1. SEM and AFM images

The SEM and AFM analysis images of each group are shown in Fig. 7. The surface of the control group can be seen strip shallow grain polished by water sandpaper, and the surface smoothness is high. No obvious cracks and depressions, only a few bumps can be seen. The surface of the Sb group can be seen with irregular pit structure and scattered strip scratches, presenting a relatively rough topography image. The surface morphology of Y-TZP in AP group and CP group was basically the same as that in blank group, and no obvious surface morphology changes occurred.

3.1.2. Surface roughness (Ra) values

Ra is the roughness symbol, and the magnitude of the Ra value corresponds to the surface roughness and smoothness of the Y-TZP specimen. The roughness values were 5.03 ± 0.57 nm for the Control group, 25.77 ± 0.71 nm for the Sb group, 5.05 ± 0.70 nm for the AP group, and 4.88 ± 0.69 nm for the CP group. The Sb group was significantly higher than all the remaining groups in roughness values, and the difference was significant ($p < 0.05$). There was no significant difference in the surface roughness of Y-TZP regardless of the plasma treatment with any gas ($p > 0.05$)(Table 1).

3.1.3. Energy-dispersive X-ray spectroscopy

The carbon and oxygen content and C:O ratio of the Y-TZP specimens are shown in Table 2 after sandblasting and plasma treatment. The C:O ratio of the Control group was 0.81 ± 0.01. After sandblasting, the Sb group was 0.56 ± 0.02. The oxygen content increased significantly, and the carbon content decreased significantly after plasma treatment. The C:O ratios of the AP and CP groups were 0.39 ± 0.02 and 0.29 ± 0.01, respectively, with the CP group significantly higher than all the remaining groups, and the differences were significant significance ($p < 0.05$)(Fig. 8).

3.1.4. Contact angle

Representative contact angle results of each group are shown in Fig. 9. 69.98 ± 1.76°, 53.96 ± 1.96°, 20.99 ± 1.52°, and 17.83 ± 1.71° for the Control, Sb, AP, and CP groups, respectively. The untreated Y-TZP surface exhibited significant hydrophobicity. However,

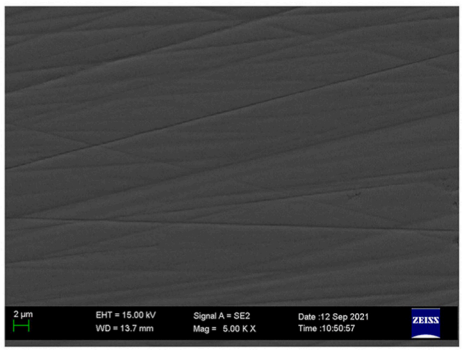
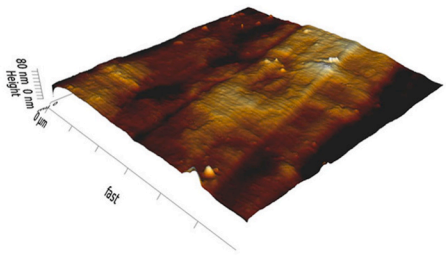
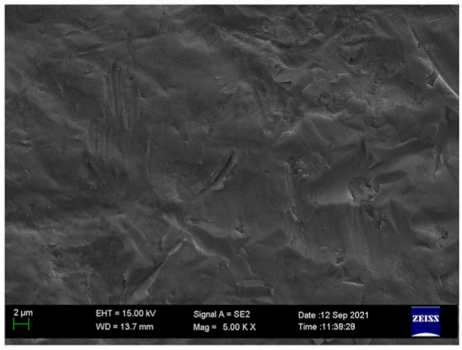
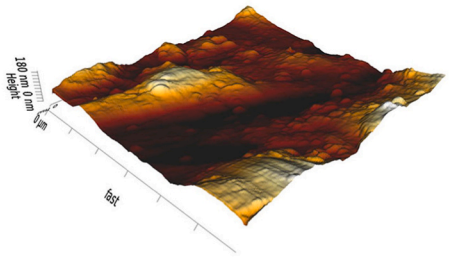
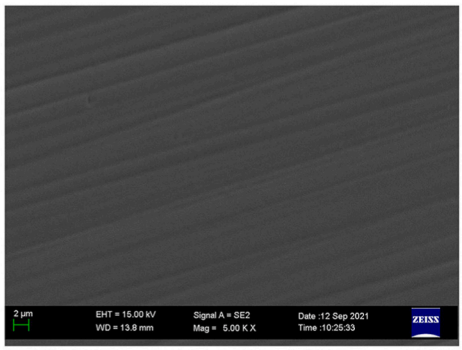
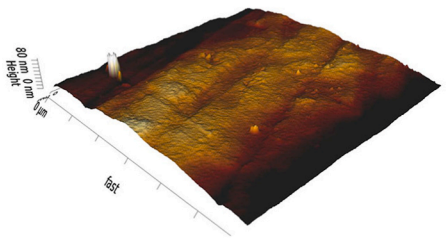
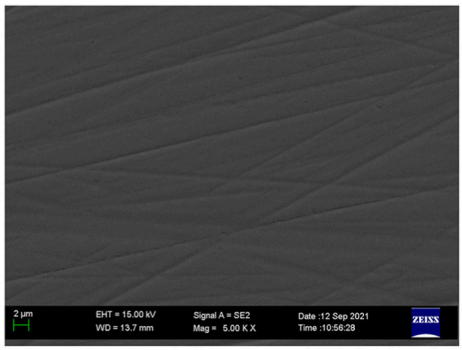
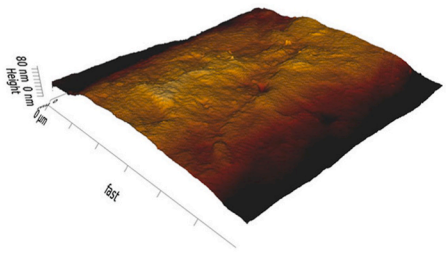
Group	SEM	AFM
Control		
Sb		
AP		
CP		

Fig. 7. SEM and AFM observation images of Y-TZP specimens (× 5000).

Table 1
Surface roughness (Ra) values of Y-TZP specimens.

Group	Surface roughness ($\bar{x} \pm s$, nm)
Control	5.03 ± 0.57
Sb	25.77 ± 0.71
AP	5.05 ± 0.70
CP	4.88 ± 0.69

Table 2
The carbon and oxygen content and C:O ratio of Y-TZP specimens.

Group	Carbon($\bar{x} \pm s$, %)	Oxygen($\bar{x} \pm s$, %)	C:O
Control	33.07 ± 1.14	41.02 ± 1.16	0.81 ± 0.01
Sb	28.86 ± 1.42	51.87 ± 1.13	0.56 ± 0.02
AP	21.07 ± 1.15	53.72 ± 0.96	0.39 ± 0.02
CP	16.85 ± 0.92	57.21 ± 1.12	0.29 ± 0.01

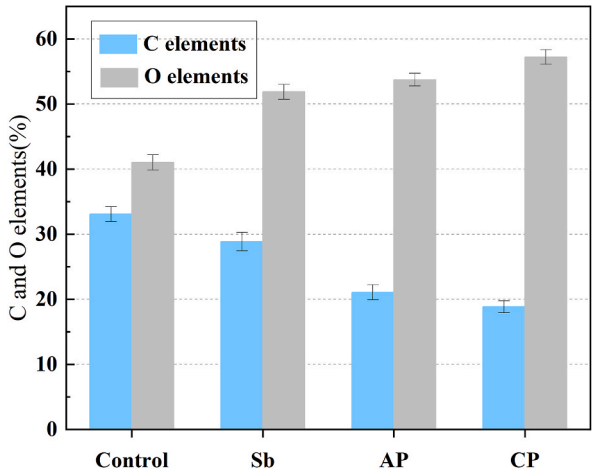


Fig. 8. Percentage of carbon and oxygen content of Y-TZP specimens in each group. Control, No surface treatment; Sb, Sandblasting; AP, Ar NTP; CP, 20 % O_2+80 % Ar combination NTP.

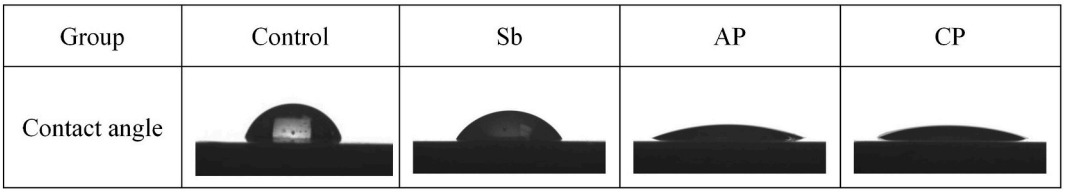


Fig. 9. Contact Angle images of each Y-TZP specimens. Control, No surface treatment; Sb, Sandblasting; AP, Ar NTP; CP, 20 % O_2+80 % Ar combination NTP.

Table 3
The contact angle of Y-TZP specimens.

Group	Contact angle($\bar{x} \pm s$, °)
Control	69.98 ± 1.76
Sb	53.96 ± 1.96
AP	20.99 ± 1.52
CP	17.83 ± 1.71

after plasma treatment, the distilled water almost lay flat on the Y-TZP surface. Moreover, the most significant hydrophilicity of NTP was observed for CP groups($p < 0.05$)(Table 3).

3.2. Shear bond strength test

Y-TZP specimens were tested for shear bond strength after bonding to isolated teeth, and the results are shown in Table 4. The control group, Sb group, AP group, and CP group were: 8.41 ± 1.56 Mpa, 12.39 ± 0.89 Mpa, 14.37 ± 1.56 Mpa, and 15.46 ± 1.52 Mpa, respectively. The control group was significantly lower than the other groups ($p < 0.05$). The values treated in the plasma group were significantly higher than those in the sandblasting group ($p < 0.05$). However, there was no significant difference in shear bond strength between the AP group and the CP group(Fig. 10).

3.3. Failure mode analysis

Failure modes were recorded as percentages for all groups(Fig. 11). Compared to the control group, the adhesive fracture rate was increased in both the NTP and Sb groups regardless of surface treatment. The control and Sb groups were dominated by adhesive fracture(Fig. 12a), while the AP and CP groups were dominated by mixed fracture(Fig. 12b). The cohesive fracture occurring in the resin cement was not observed for all groups.

4. Discussion

In previous studies, it has been shown that the treatment with NTP improves the SBS of Y-TZP to resin cement [15]. Therefore, the purpose of this study was to evaluate the effect of NTP treatment with different gases as media on the surface energy of Y-TZP and their SBS to resin cement. The first null hypothesis was rejected since the different treatments improved the surface energy of the Y-TZP surfaces and increased the SBS to the self-adhesive resin cement. Additionally, there was no statistically significant difference in the SBS of Y-TZP with self-adhesive resin cement due to NTP treatment with different gases. However, the surface energy of the CP group was significantly higher than that of the AP group, leading to the partial acceptance of the second null hypothesis.

Okutan et al. [18] showed that sandblasting zirconia increased the roughness and improved the bond strength of resin cement to zirconia while creating microcracks on the surface of the zirconia. In this experiment, the surface of the Sb group showed a large number of irregular bumps and obvious uneven mound-like patterns, and the roughness increased significantly. Sandblasting affects the morphology of the zirconia surface with structural defects, leading to an increase in the grain crystalline phase transition from tetragonal to monoclinic, which significantly impacts the mechanical properties and long-term stability of zirconia [10]. The AFM 3D images of the AP and CP groups were similar to the control group, with high surface flatness and only a few bumps visible, with no significant differences between the roughness values. Zens et al. [19] reported that chemical treatment could produce a nano-modification effect on the zirconia surface with less damage caused to its surface. Chan et al. [20] and Zheng et al. [21] showed that the treatment of zirconia with plasma using Ar, He, 95 % Ar combined with 5 % O_2 as working gas revealed no significant change in surface roughness. This is consistent with the results of the current study.

Ito et al. [22] attributed the presence of carbon on the surface of zirconia to the adsorption of carbonaceous organic matter on the surface of zirconia in the atmosphere. The oxygen content of the Sb group was 51.87 ± 1.13, and the carbon content was 28.86 ± 1.42, with a slight decrease in carbon compared to the control group. Previously reported literature stated [23,24] that sandblasting zirconia produces a roughening effect along with a cleaning effect that breaks the C–C bond on the zirconia surface and becomes one of the treatments for cleaning the contaminants on the zirconia surface.

The elemental carbon content on the surface of zirconia in the AP and CP groups treated with plasma decreased significantly, and the elemental oxygen content increased significantly, with significant differences between the two groups. An electric field is applied to the neutral gas to generate plasma. During the plasma action, the activation of water molecules in the ambient air into the gas phase generates electrons and ions, and collisions with the zirconia surface promote the amount of –OH. The XPS analysis studied by Henningsen et al. [25] confirmed the formation of oxygen functional groups on the surface of zirconia after treatment with ionomers. In our study, the contact angle of the plasma group was significantly lower than that of the control and Sb groups, indicating that the hydrophilicity of the zirconia surface increased significantly after plasma treatment, and the surface modification of zirconia was better than that of sandblasting. Moreover, the contact angle of the CP group was significantly lower than that of the AP group. It has been reported [26] that plasma treatment increases the formation of reactive peroxide radicals and excess functional groups (e.g., C–O and C–OH) on the surface of inert materials such as zirconia, resulting in significant activation of the material surface. These activated ions and molecules can modify or induce various chemical and physical reactions on the zirconia surface. The purification of organic impurities and the promotion of water physical adsorption significantly improve the zirconia surface wettability and enhanced hydrophilicity without overly changing the overall properties of the substrate [27,28]. Studies have reported Ar as an inert gas with low breakdown voltage, and mixing some reactive gases such as O_2, He, or air in small amounts into the inert gas can produce more chemically active substances such as O_3, H_2O_2, NO, and –OH at low temperatures as the breakdown voltage decreases [29]. Zheng et al. [21] showed that when zirconia surfaces were subjected to atmospheric pressure cold plasma treatment, the plasma produced by mixing Ar and O_2 introduced a more significant excess of highly reactive oxygen radicals on the material surface. Compared to Ar plasma, the mixed gas plasma had an elevated elemental oxygen content, a smaller contact angle, and a significant increase in wettability resulting in a significant increase in the early adhesion of human gingival fibroblasts. These reports support our findings.

In our experiments, comparing the control and Sb sandblasted groups, the shear bond strength was significantly higher in both

Table 4
Shear bond strength (SBS) of Y-TZP specimens.

Group	Shear bond strength($\bar{x} \pm s$, MPa)
Control	8.41 ± 1.56
Sb	12.39 ± 0.89
AP	14.37 ± 1.56
CP	15.46 ± 1.52

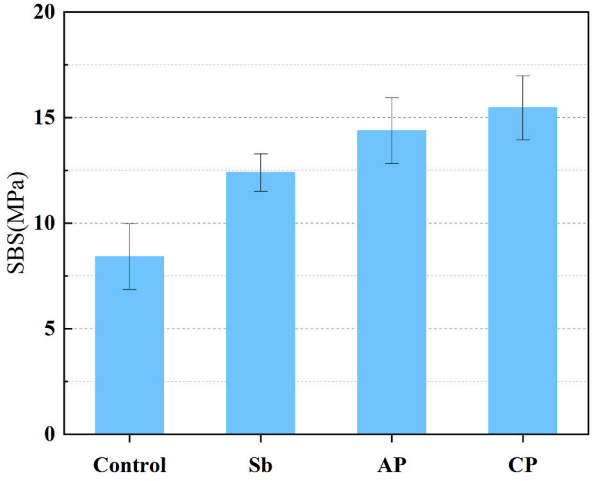


Fig. 10. Shear bond strength (SBS) of each group of Y-TZP with different surface treatment methods. Control, No surface treatment; Sb, Sandblasting; AP, Ar NTP; CP, 20 % O_{2+}80 % Ar combination NTP.

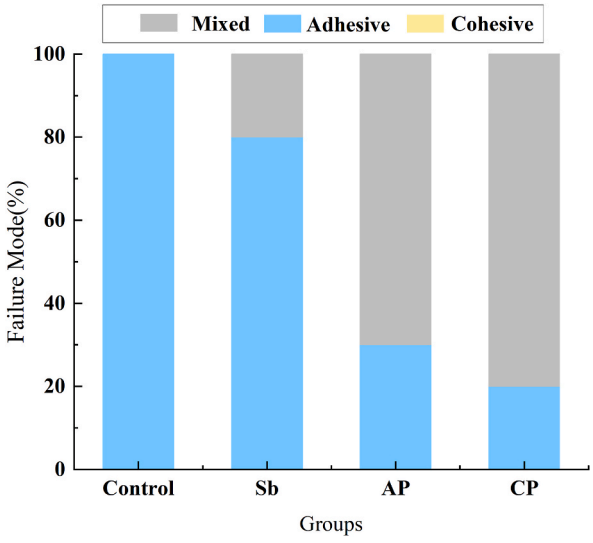


Fig. 11. Percentage of failure mode of Y-TZP specimens in each group. Control, No surface treatment; Sb, Sandblasting; AP, Ar NTP; CP, 20 % O_{2+}80 % Ar combination NTP.

plasma-treated groups, and the difference was statistically significant. Failure Mode was observed for both AP and CP groups, and both were dominated by mixing damage. Simultaneous zirconia and resin cements could be observed, with significant differences compared to the blank and sandblasted groups, which were dominated by adhesive damage. These indicate that plasma treatment of zirconia can increase the shear bond strength between zirconia and resin cement agent. Canullo et al. [30] showed that plasma-induced electronic oxidation of H_2O produces dissociation of –OH and H+, leading to elevated surface energy and producing a super hydrophilic effect. Surface energy (SE) is a term indicating the intermolecular forces on the surface of a material, which depends on the values of the surface polar and dispersed components [31]. A common way to reflect the size of SE is to measure the contact angle of the liquid on

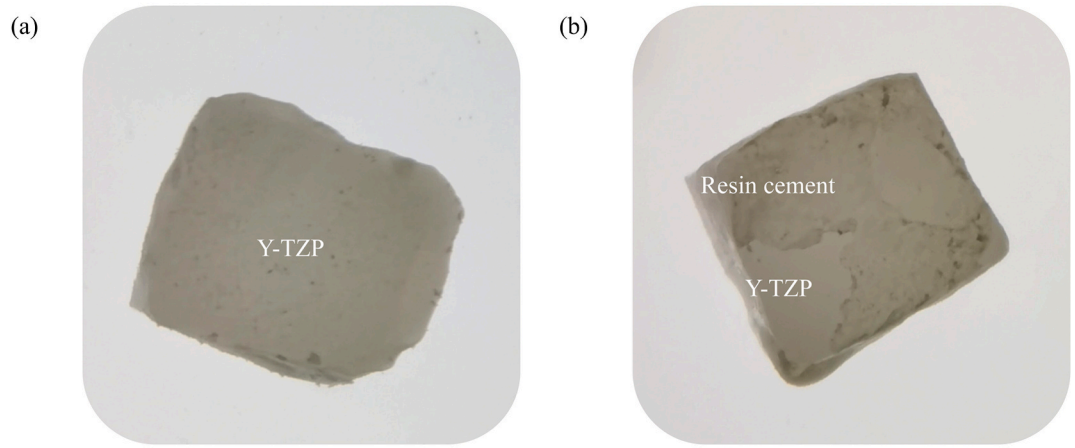


Fig. 12. Fracture mode images of Y-TZP specimens. (a) Adhesive fracture. (b) Mixed fracture.

the material surface, the amount of which is inversely related to SE. The smaller the contact angle, the larger the SE value, the better the wettability of the surface and the better the adhesive properties of the material. In the previous study, the CP group had the lowest contact angle, while the shear bond strength was higher than that of the AP group. However, there was no significant difference between the two groups. Compared to plasma from other gases, Ar plasma requires lower energy to excite high-energy electrons and ions, generating less heat and having an insignificant effect on zirconia grain size. At the same time, Ar plasma increases the polar component by adding oxygen elements and reducing carbon-based contaminants, producing a super hydrophilic surface [32]. It has also been reported [33] that the ionization energy of Ar used for plasma irradiation is about 15.8eV, and the presence of this energy may affect the bond strength. Mixtures of molecular gases with chemically inert gases such as He, Ne, and Ar can modify the plasma discharge kinetics and show better biological properties [29]. It was shown [34] that in a plasma generated by a gas mixture, the excited inert gas can ionize the reactive gas by collision through energy transfer, which leads to changes in the discharge characteristics and is more favorable to improving the shear bond strength. Another study [35] showed that O_2 plasma treatment could introduce oxygen to the surface of inert ceramic materials with dense structures and subsequently promote the formation of reactive superoxide radicals(R-*O*-O-), which induce chemical substance alterations and thus reduce shear bond strength. There was no significant difference in shear bond strength between AP and CP groups in this experiment, probably because of the mixing ratio of Ar and O_2 in the plasma.

The effectiveness of applying low-temperature atmospheric pressure plasma depends mainly on the experimental parameters, including gas, power, time, and gas flow rate, the most important of which is the type of gas [36]. One of the limitations of this experiment is that only the plasma with 20 % Ar + 80 % O_2 mixture was studied. The Ar and O_2 mixing ratio should be adjusted in subsequent experiments. The plasma treatment time and power should be improved to further verify the plasma treatment parameters with the best effect and improve the bonding performance of zirconia to a greater extent. Another limitation is that only short-term bond strength tests were conducted in this experiment and no thermal cycling procedure was performed. It has been reported that [37] the bond durability effect of plasma-treated zirconia was not satisfactory, probably because impurities were deposited on the ceramic surface during the plasma treatment, which affected the long-term bonding effect. To improve the comprehensiveness and credibility of the experiments, subsequent thermal cycling experiments should be conducted to test the shear bond strength of plasma-treated zirconia after simulated artificial ageing tests. This will allow for observation of the long-term bonding effect of plasma treatment.

5. Conclusions

This experiment showed that plasma treatment could effectively increase the shear bonding properties of zirconia and has a good application in the bonding process of clinical zirconia restorations. Both AP (argon NTP) and CP (20 % oxygen and 80 % argon combination NTP) significantly increased the shear bond strength between zirconia and resin cement compared to sandblasting. Moreover, the surface morphology after plasma treatment was similar to that before treatment and did not significantly change the surface properties of zirconia.

Data availability statement

Has data associated with your study been deposited into a publicly available repository? No.
Please select why. Please note that this statement will be available alongside your article upon publication.
Data included in article/supp. material/referenced in article.

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CRediT authorship contribution statement

Yulin Jiang: Writing – original draft. **Xudong Bao:** Data curation. **Yang Yu:** Data curation. **Yannan Zhang:** Methodology. **Min Liu:** Validation. **Fanhao Meng:** Software. **Bo Wang:** Supervision. **Jianfeng Chen:** Writing – review & editing.

Declaration of competing interest

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

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