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RESEARCH AND EDUCATION

Effect of different times of nonthermal argon plasma treatment on the microtensile bond strength of self-adhesive resin cement to yttria-stabilized tetragonal zirconia polycrystal ceramic

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ABSTRACT

Statement of problem. Nonthermal argon plasma may increase the surface energy of yttria-stabilized tetragonal zirconia polycrystal (Y-TZP) dental ceramics. However, studies that evaluated the effect of increased plasma treatment times on the bond strength of resin cements to Y-TZP ceramics are lacking.

Purpose. The purpose of this in vitro study was to evaluate the effect of different nonthermal argon plasma (NTAP) treatment times on the surface energy and bond strength of a self-adhesive resin cement to Y-TZP ceramic.

Material and methods. Forty-eighty Y-TZP plates were divided into 2 groups (n=24): as-sintered (AS) and airborne-particle abrasion (APA) with 50-μm Al_2O_3, which were subdivided into 4 groups (n=6) according to the time of NTAP treatment: 0, 20, 60, and 120 seconds. The surface energy was evaluated with a goniometer. Forty Y-TZP blocks submitted to the same surface treatments (8 groups; n=5) were cemented to composite resin blocks, using a self-adhesive resin cement. After storage in distilled water at 37°C for 24 hours, the Y-TZP-composite resin blocks were cut into beams and submitted to a microtensile bond strength (μTBS) test. Data were analyzed using 2-way ANOVA and the Tukey honestly significant differences test (α=.05).

Results. Treatment with NTAP increased the surface energy for AS and APA groups (P<.05). For both groups, the μTBS was as follows: 0 seconds < 20 seconds < 60 seconds = 120 seconds (P<.05). Only after 120 seconds of NTAP treatment was the μTBS of APA higher than that of AS (P<.05).

Conclusions. Treatment with NTAP improved the surface energy and increased the μTBS of self-adhesive resin cement to Y-TZP ceramic, with higher times of plasma treatment resulting in higher bond strength. (J Prosthet Dent 2019;121:485-91)

Because of its improved mechanical properties and suitable optical behavior, yttria-stabilized tetragonal zirconia polycrystal (Y-TZP) ceramic has been popular for the fabrication of crowns, fixed dental prostheses, and dental implants.[1,2] Y-TZP dental ceramic is composed of 100% metastable tetragonal, *t*, zirconia phase, retained at room temperature by the careful control of the grain size (<0.5 μm) and the presence of a stabilizer (Y_2O_3, 2-5 mol%).[3,4] This microstructure favors the development of a toughening mechanism that strengthens the ceramic. However, the absence of a dioxide glass phase makes Y-TZP ceramics resistant to hydrofluoric acid,[5] which is the

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Clinical Implications

Treatment of nonreactive Y-TZP ceramic surface with increased nonthermal argon plasma times is a promising protocol for improving its interaction with self-adhesive resin cements.

protocol most commonly used to prepare the intaglio surface of ceramic restorations for bonding.[1,2] To overcome this limitation, different protocols such as airborne-particle abrasion, tribochemical silica coating, use of phosphate acidic monomers, use of functional silane monomers, vitrification, and silica nanoparticle surface deposition have been proposed to achieve reliable and long-lasting interaction between Y-TZP ceramics and resin cements.[6-20] Unfortunately, none of these approaches has resulted in a definitive protocol for the adhesion of resin cements to Y-TZP ceramics.

Treating the Y-TZP ceramic with nonthermal plasma has shown promising results for increasing bonding with resin cements.[21-23] Nonthermal gas plasma technology is based on a partially ionized gas containing highly reactive species such as electronically excited atoms, molecules, electrons, ions, and free radicals at near room temperature. These structures can react with the material surface and modify it physically and/or chemically, improving surface energy and wettability, without affecting bulk properties.[24,25] Argon plasma treatment significantly increases the surface energy of Y-TZP dental ceramics.[22,26,27]

These nonthermal argon plasma (NTAP) studies used relatively short treatment times. Therefore, an investigation of longer NTAP treatment times on the interaction between Y-TZP ceramics and resin cements was warranted. The purpose of this study was to evaluate the influence of different NATP treatment times on the surface energy and bond strength of a self-adhesive resin cement to Y-TZP. The null hypotheses tested were that treatment with NTAP does not influence the surface energy of the Y-TZP and that the bond strength between this material and a resin cement and the time of NTAP treatment do not affect the surface energy of the Y-TZP and the bond strength between this material and a resin cement.

MATERIAL AND METHODS

A Y-TZP ceramic (Lava Frame; 3M ESPE) was used to investigate the hypotheses of the present study. For surface energy (SE) evaluation, Lava blocks were cut with a diamond blade (Series 15LC #114254; Buehler), using a precision sectioning machine (IsoMet 1000; Buehler) at 800 rpm, producing 48 ceramic plates (5.8×5.8×1.4 mm). After plates were sintered according to the manufacturer's instructions, they were ultrasonically cleaned in distilled water for 5 minutes and divided into 2 groups (n=24): those maintained as-sintered (AS) and those that were airborne-particle abraded (APA) with 50 μm Al_2O_3 particles at a pressure of 280 kPa for 12 seconds at 10 mm. Afterwards, these 2 groups were ultrasonically cleaned in 96% ethanol for 5 minutes and subdivided into 4 groups (n=6), according to the NTAP treatment time: 0, 20, 60, and 120 seconds.

The NTAP treatment was performed by using an experimental glass reactor consisting of a tube of 5 cm diameter and 80 cm long, evacuated by means of a mechanical pump to a pressure lower than 5 Pa. Gas was allowed to fill the reactor up to a pressure of 10 Pa. Nonthermal plasma was generated within the glass cylinder under vacuum by the action of an induced magnetic field from the current passing through an electrical coil surrounding the cylinder. Y-TZP ceramic surfaces were treated with argon gas at 60 W for 0, 20, 60, or 120 seconds. At the end of this process, radio frequency was turned off before the specimens were exposed to air.

The Owens-Wendt-Rabel-Kaelble method was used for SE determination.[28] The contact angle between the Y-TZP plate surfaces and 2 liquids with different polarities (water [polar] and diiodomethane [apolar]) were carried out with a goniometer (model 100; Ramé-Hart Instrument Co). Forty measurements at an interval of 1 second were made for each Y-TZP plate. Three plates (n=3) were used per liquid for each group. Based on these measurements, the Ramé-Hart software calculated the SE and the polar and dispersive components for all groups.

Three Y-TZP plates submitted to each condition (AS and APA) were analyzed using atomic force microscopy (XE7; Park Systems). Areas of 10×10 μm were scanned in contact mode, using a probe exhibiting a tip radius of curvature <10 nm, a scan rate of 1 Hz, and a set point of 1.16 nN. The topography was evaluated with XEP software with a 3D view.

The experimental arrangement of the microtensile bond strength (μTBS) testing is illustrated in Figure 1. Forty Y-TZP ceramic blocks (7.6×5.5×4.4 mm), obtained exactly as those used to measure the SE, were duplicated to build up composite resin blocks (Z100; 3M ESPE) with dimensions similar to the Y-TZP blocks. The composite resin blocks were fabricated in 5 increments, individually light polymerized for 40 seconds with an irradiance of 600 mW/cm^2 (Optilux 501; Demetron). Top surfaces of the composite resin blocks were polished with 600-grit SiC paper to standardize the roughness.

Immediately before cementation, the Y-TZP blocks were divided into 8 groups (n=5) according to surface treatment (AS and APA versus NTAP treatment time [0, 20, 60, and 120 seconds]). The Y-TZP ceramic blocks were cemented to the composite resin blocks with self-adhesive resin cement (RelyX U200; 3M ESPE). Resin cement was applied to the ceramic surface, and the

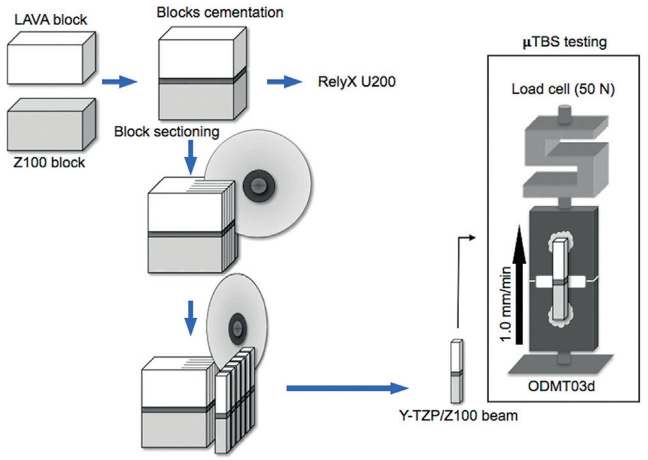

Figure 1. Experimental design of microtensile bond strength testing (μTBS). ODM, the μTBS device; Y-TZP, yttria-stabilized tetragonal zirconia polycrystal.

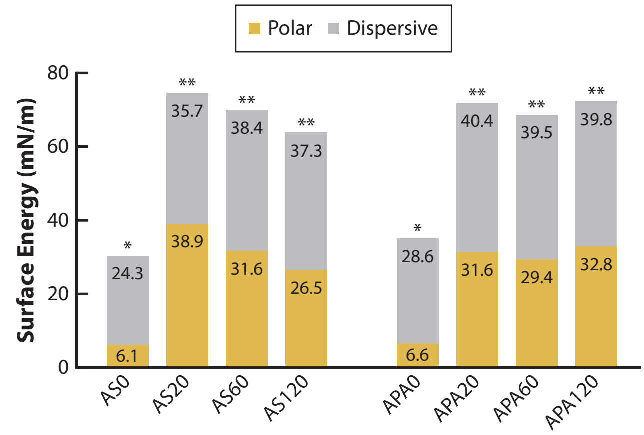

Figure 2. Means of polar and dispersive components of surface energy for all groups. Bars with same number of asterisks statistically similar (*P*>.05). APA, airborne-particle abraded; AS, as-sintered.

composite resin block was placed and held under a load of 5.9 N for 5 minutes at room temperature (23 ±1°C). After excess cement was removed, the resin cement was light polymerized for 40 seconds on each side of the Y-TZP ceramic-composite resin block with an irradiance of 600 mW/cm^2.

After Y-TZP ceramic-composite resin blocks were stored in distilled water at 37°C for 24 hours, they were longitudinally sectioned across the bonded interfaces in both x and y axes, producing beams with a cross-sectional area of approximately 1 mm^2 and 10 mm in length. Light-body silicone impression material (Perfil; Coltène) was injected between the slices to absorb the vibration generated during the second cut. Each beam was attached to a μTBS device (ODMT03d; Odeme Dental Research) with cyanoacrylate adhesive and loaded in tension with a 50-N load cell at a crosshead speed of 1.0 mm/min, using a universal testing machine (DL 2000; EMIC) until failure occurred. The μTBS (MPa) was obtained by dividing the load at failure (N) by the cross-sectional area of the beam (mm^2).

Each failed beam was evaluated using stereomicroscopy at ×40 magnification, and the failure mode was classified as adhesive (failures at the adhesive interface), cohesive (failures occurring mainly within Y-TZP ceramic, resin cement, or composite resin), or mixed (mixture of adhesive and cohesive failure within the same fractured surface). Additionally, beams presenting different failure modes and with the μTBS value close to the mean of each group were selected and observed with scanning electron microscopy (SEM) (PhenomProX; PhenomWorld) operating in the backscattered mode in a low vacuum environment. The SEM images were made at a magnification of ×4000.

Data were analyzed using statistical software (Statgraphics Centurion XVI; STATPOINT Technologies Inc). Sample size for each dependent variable (μTBS and SE) was calculated based on mean ±SD values obtained in the respective pilot study. Prematurely failed beams were included in the statistical analysis by assigning a value that corresponded to half of the minimum μTBS for their experimental group, and the specimens that presented cohesive failures were excluded.[29,30] After the normal distribution of errors was evaluated using the Shapiro-Wilk test and homoscedasticity of variances with the Levene test, SE and μTBS data were analyzed using 2-way ANOVA and Tukey honestly significant differences post hoc test. Regression analysis was performed to investigate the correlation between μTBS and NTAP treatment times (α=.05).

RESULTS

Figure 2 shows the mean values (mN/min) of the polar and dispersive components of SE (the sum of these 2 values represents the absolute value of SE). The SE significantly increased after NTAP treatment (*P*<.05), with this increase being mostly influenced by the polar component. The NTAP treatment time did not affect the SE results (*P*>.05).

Representative atomic force microscopy images of the Y-TZP ceramic surfaces are shown in Figure 3. Zirconia grain limits can be easily identified in AS specimen (Fig. 3A). The APA specimen shows deep valleys (white arrows) without the presence of zirconia grain limits (Fig. 3B).

Results of 2-way ANOVA showed that μTBS was affected by NTAP treatment time (*P*<.05). The means of μTBS are shown in Table 1. In specimens not treated with NTAP, APA presented significantly higher values for μTBS than for AS (*P*<.05). For both AS and APA, treatment with NTAP significantly increased the μTBS: 0 seconds<20 seconds<60 seconds=120 seconds (*P*<.05).

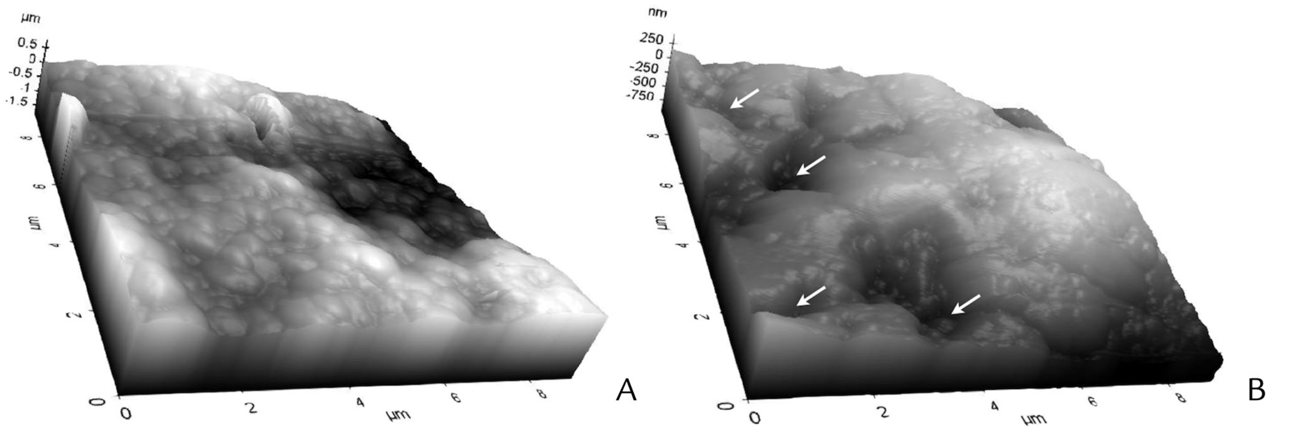

Figure 3. Representative atomic force microscopy image of yttria-stabilized tetragonal zirconia polycrystal ceramic surfaces after surface treatments. A, As-sintered surface. B, Airborne-particle abraded surface. Scanned area was 9.09×9.09 µm. The white arrows show deep valleys produced by air particle abrasion in the the ceramic surface.

Only after 120 seconds of NTAP treatment was the µTBS of APA significantly higher than that of AS ($P<.05$). Regression analysis showed a strong positive correlation between µTBS and NTAP treatment times (R=0.80; P=.025) (Fig. 4). The number of pretesting failures and number of beams submitted to µTBS testing are summarized in Table 2. AS0 presented the highest number of pretesting failures, followed by APA0, AS20, APA20, and AS120, whereas for the groups AS60, APA60, and APA120, no beam underwent premature failure. The failure mode distribution is shown in Figure 5. The groups not treated with NTAP showed a predominance of adhesive failures (94% for AS and 87% for APA). Only AS20 showed a small percentage (8.3%) of cohesive failures. An increase, ranging from 25% for AS20 to 61% for APA120, was found in the percentage of mixed failures after treatment with NTAP. Representative SEM micrographs of debonded specimens are shown in Figure 6.

Table 1. Mean ±standard deviation µTBS (MPa)

Surface Treatment	Time of NTAP Treatment (s)			
	0	20	60	120
AS	6.2 ±0.7^{Aa}	20.6 ±3.6^{Ab}	31.5 ±4.1^{Ac}	31.0 ±2.2^{Ac}
APA	11.6 ±0.9^{Ba}	27.2 ±2.4^{Ab}	36.7 ±4.9^{Ac}	44.2 ±7.8^{Bc}

µTBS, microtensile bond strength; NTAP, nonthermal argon plasma. Means followed by different superscript letters (uppercase-column, lowercase-row) indicate statistical differences (Tukey test, $P<.05$).

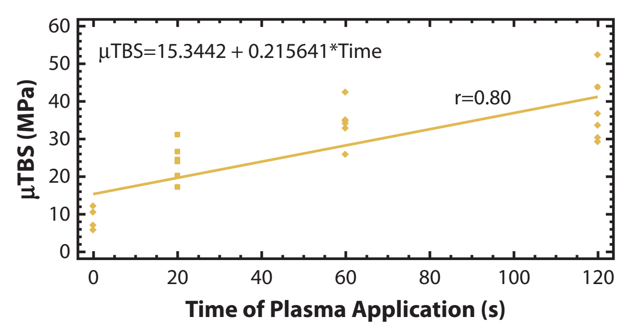

Figure 4. Linear regression line of microtensile bond strength (µTBS) plotted against nonthermal argon plasma treatment time (R=0.80/ µTBS=15.34 + 0.22s), P=.025.

DISCUSSION

Previous studies have demonstrated that the treatment of nonreactive zirconia ceramics with NTAP increases their bonding with resin cements.[21,22] Based on this, the purpose of the present study was to investigate the effect of longer NTAP treatment times on Y-TZP ceramic bonding to resin cements. Because the treatment with NTAP improved the SE of the Y-TZP ceramic surfaces and increased the bonding with self-adhesive resin cement, the first null hypothesis was rejected. In addition, although the different NTAP treatment times resulted in similar SEs, the bond strength of Y-TZP to self-adhesive resin cement after 60 and 120 seconds was statistically higher than that after 20 seconds. Thus, the second null hypothesis was partially accepted.

The methacrylate phosphate monomers present in self-adhesive resin cements are capable of establishing chemical interactions with the hydroxyl groups of the passive coating of ZrO_2 present in Y-TZP ceramics.[8,14,15] Hence, this type of resin cement performs better than conventional resin cements in terms of bonding to Y-TZP ceramics.[5,11,14] This was why RelyX U200 was used in the present study. The purpose was to investigate the effect of different NTAP treatment times on bond strength to Y-TZP ceramic with the best available luting system.

The SE is a good predictor of the level of intermolecular interactions between 2 materials as it is directly related to the hydrophilicity and wettability of the involved surfaces. To achieve suitable adhesion, the adherent (or solid) should present higher SE than the

Table 2. Total beams produced, number and percentage of pretesting failures, and total beams submitted to µTBS test

Group	Total Beams	Number of Pretesting Failures (%)	Number of Beams Submitted
AS0	80	23 (28.8)	57
AS20	80	11 (13.8)	69
AS60	80	0 (0)	80
AS120	80	7 (8.8)	73
APA0	80	16 (20.0)	64
APA20	80	9 (11.3)	71
APA60	80	0 (0)	80
APA120	80	0 (0)	80

AS, as-sintered; APA, airborne-particle abrasion with 50-µm Al_2O_3; µTBS, microtensile bond strength.

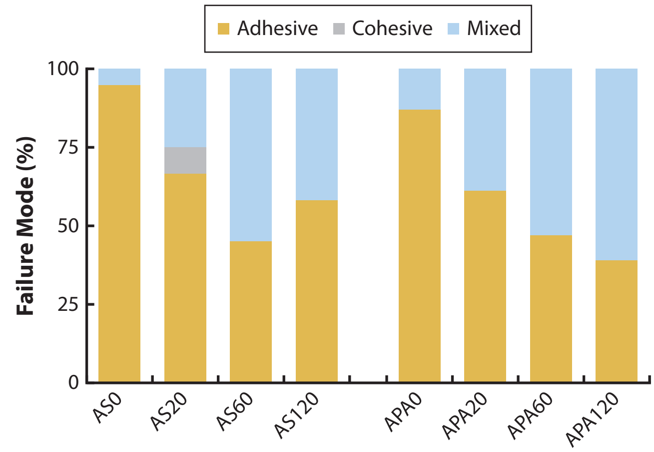

Figure 5. Failure mode distribution for all groups. APA, airborne-particle abraded; AS, as-sintered.

surface tension of the liquid (or adhesive), which means that the liquid will properly spread over the solid, thereby creating a better interaction. Regardless of the previous surface condition (AS or APA), NTAP increased the SE of Y-TZP surfaces, with this increase being strongly influenced by the polar component**.** This finding agrees with those of other studies.[22,26,27] However, a significant influence was not found for different NTAP treatment times on SE (Fig. 2). This result is supported in the study by Silva et al,[26] who found no significant differences in the SE of a Y-TZP ceramic after NTAP treatment for 5, 10, or 20 seconds. Valverde et al[22] showed an increase in the O level (XPS analysis) along with an improvement of the SE (∼60 mN/min) in AS and APA Y-TZP ceramic surfaces treated with NTAP for 10 seconds. These studies showed that the polar component of SE increased by 247% for an AS surface and by 57% for an APA surface. In the present study, after 20 seconds of NTAP, the increase of polar component was 538% and 379% for AS and SB groups, with the surface energy reaching values of 74.6 mN/m and 72.0 mN/m, respectively. Thus, it was hypothesized that after 20 seconds of treatment, the NTAP would produce an increase in the level of O and that this would lead to a higher SE, which would remain stable after longer times of treatment (60 seconds and 120 seconds).

The values of µTBS for non-NTAP-treated groups (AS=6.2 MPa and APA=11.6 MPa) are in good agreement with those of other reports.[14,15] Also, APA showed a significantly higher µTBS than the AS group. This result could be explained by the additional microretention of the resin-cement into the grooves produced by the 50 µm Al_2O_3 particles in Y-TZP surfaces (Fig. 3). After NTAP treatment for 20 seconds, µTB increased 232% (6.2 to 20.6 MPa) for AS and 134% (11.6 to 27.2 MPa) for APA. Furthermore, after NTAP treatment for 60 seconds, µTBS increased 408% for AS and 216% for APA, without any statistically significant differences. Interesting comparisons can be made with the results of Valverde et al,[22] who reported a µTBS increase of 134% for the AS surface and 115% for APA surface, after 10 seconds of NTAP treatment with a hand-held unit. First, the results of both studies show that the action of NTAP was more pronounced in AS than in APA surfaces. Moreover, in the present study, the µTBS for the AS and APA groups were statistically similar after NTAP treatment for 20 seconds. These aspects could be considered advantageous for the protocol of NTAP treatment in AS Y-TZP ceramic surfaces. Although airborne-particle abrasion is able to increase the interaction between Y-TZP ceramics and resin cements, other studies have shown that, depending on the size of Al_2O_3 particles, this protocol may also produce subsurface damage and microcracks, which might negatively affect the mechanical properties and reliability of Y-TZP ceramics.[3,4,9-11,14] Therefore, if the treatment of AS Y-TZP ceramic surface with NTAP produces values of bond strength comparable with those obtained in NTAP-treated APA surfaces, the first protocol could be a good option for the cementation of Y-TZP ceramic restorations.

A recent study demonstrated that an experimental adhesive system showed increased conversion in the region closer to the dentin treated with NTAP for 30 seconds than in the dentin that was not submitted to plasma. This behavior was explained by plasma-induced polymerization.[25] This phenomenon occurs when the energy from collisions of excited particles (direct transfer) and the irradiation of photons (indirect transfer) are transferred to the matter submitted to nonthermal plasma.[31] According to these authors, the energy transferred from the plasma to the dentin immediately coated with the experimental adhesive remained active and induced the cleavage of methacrylate C=C bonds of the adhesive monomers at the dentin surface, forming diradicals that contributed to the adhesive polymerization reaction. This explanation supports the findings of Chen et al,[32] who showed that an experimental, photoinitiator-free, self-etch adhesive polymerized with nonthermal

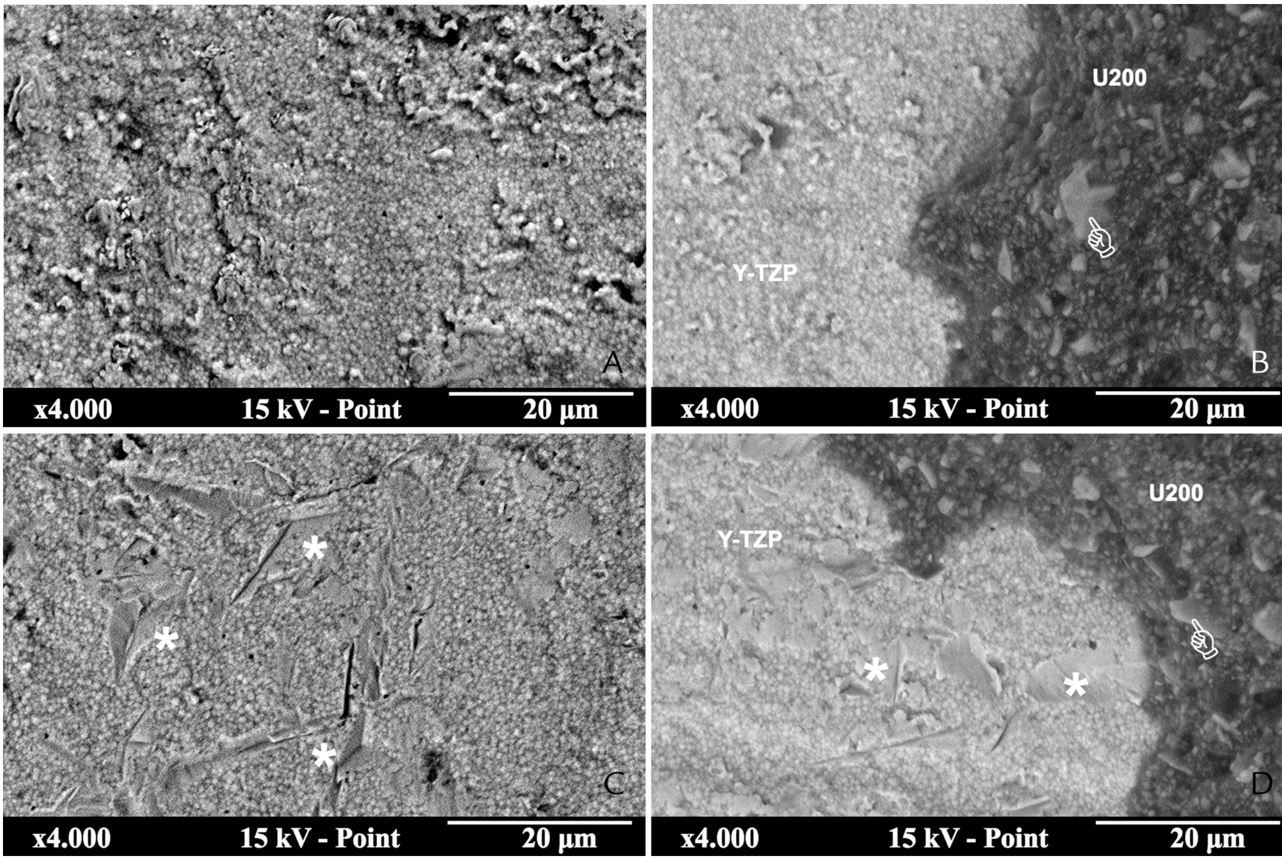

Figure 6. Representative scanning electron microscopy micrographs of debonded specimens. A, AS0, adhesive failure. B, AS60, mixed failure. C, APA60, adhesive failure. Grooves produced by airborne-particle abrasion (white asterisks) can be seen in the Y-TZP ceramic surface. D, APA120, mixed failure (original magnification ×4000). B, D, White arrows show filler particles protruding from the fractured surfaces of self-adhesive resin cement. APA, airborne-particle abraded; AS, as-sintered.

argon plasma for 40 seconds showed a higher degree of conversion than its light-polymerized counterpart. Thus, the higher μTBS presented by the APA group after treatment with NTAP for 120 seconds (44.2 MPa) might also have been influenced by the plasma-induced polymerization. This could have cleaved the C=C bonds of RelyX U200 near the greater APA Y-TZP surface, increasing its degree of conversion and favoring the interaction at the adhesive interface. Furthermore, the high SE of the APA group after NTAP treatment for 120 seconds might also have increased their interaction, through Van der Waals forces, with the phosphate groups of the monomers present in RelyX U200, phenomena that could also have contributed to the higher bond strength presented by this group.[22]

Results of failure mode analysis and the number of pretesting failures were consistent with the μTBS values obtained in the present study, that is, without NTAP treatment, AS and APA surfaces showed predominantly adhesive failures and a higher number of pretesting failures associated with low values of μTBS.[14] Differently, after NTAP treatment, a higher percentage of mixed failures, along with an increase in μTBS, was observed. This behavior is supported by other investigators and can be interpreted as the bond strength having matched the cohesive strength of RelyX U200.[11,12,14] The higher ±SD presented by APA60 and APA120 groups could be considered a result of this behavior. Also, a moderate correlation (R=–0.72; P<.05) was found between the number of pretesting failures and the NTAP treatment times. This finding by itself reinforces the fact that longer NTAP treatment times may improve the interaction between Y-TZP ceramics and self-adhesive resin cements.

Although the results of the present study showed interesting aspects regarding the influence of increased NTAP treatment times on the bonding performance of a Y-TZP ceramic, some aspects such as the use of only 1 self-adhesive resin cement and the absence of a long-term investigation are limitations. These and other aspects, such as the simulation of mastication forces, should be addressed in future investigations.

CONCLUSIONS

Based on the results of this in vitro study, the following conclusions were drawn:

1. Treatment with NTAP may improve the surface energy of Y-TZP ceramic to prepare it for bonding.
2. Up to 120 seconds, the increase in the NATP treatment time may be a good strategy for improving the bonding of Y-TZP ceramics with self-adhesive resin cements.

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