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The Effect of Sandblasting and Different Primers on Shear Bond Strength Between Yttria-tetragonal Zirconia Polycrystal Ceramic and a Self-adhesive Resin Cement

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Clinical Relevance

The combined use of air-abrasion and zirconia primer should be recommended for strong and durable bonding between yttria-tetragonal zirconia polycrystal and self-adhesive resin cement.

SUMMARY

Purpose: To evaluate the effect of zirconia primers, air-abrasion, and tribochemical surface treatment methods on the shear bond strength between yttria-tetragonal zirconia polycrystal (Y-TZP) ceramic and self-adhesive resin cement.

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Methods and Materials: Y-TZP ceramic surfaces were ground flat with 600-grit silicon carbide paper and then divided into seven groups of 10 and treated as follows: untreated (control), Monobond Plus, Z-PRIME Plus, ESPE Sil with CoJet, air-abrasion, Monobond Plus with air-abrasion, and Z-PRIME Plus with air-abrasion. Self-adhesive resin cement was placed onto the treated Y-TZP specimens for each group. All specimens were thermocycled and subjected to a shear bond strength test. Scanning electron microscope images of the fractured areas and x-ray diffraction (XRD) analysis of the surface-treated Y-TZP specimens were performed. Data were statistically analyzed using one-way analysis of variance and the Student-Newman-Keuls multiple comparison test ($p<0.05$).

Results: The Z-PRIME Plus treatment in combination with air-abrasion produced the highest bond strength (16.50±2.26 MPa), followed by air-abrasion (10.56±3.32 MPa), and then Monobond Plus combined with air-abrasion (8.93±3.13 MPa), ESPE Sil after CoJet application (8.54±3.98 MPa), and the Z-PRIME Plus group (8.27±2.79 MPa). The control (3.91±0.72 MPa) and Monobond Plus (4.86±1.77 MPa) groups indicated the lowest results ($p<0.05$). The XRD results showed the peaks of the monoclinic phase for the air-abrasion and CoJet treatment groups compared with the Y-TZP control.

Conclusion: Z-PRIME Plus primer application after air-abrasion presented the best results for improving the bond strength between Y-TZP ceramic and self-adhesive resin cement.

INTRODUCTION

All-ceramic restorations are a potentially more effective method compared with metal ceramic restorations in clinical situations requiring highly demanding esthetic restorations.[1] Due to its high fracture toughness and chemical durability, yttria-tetragonal zirconia polycrystal (Y-TZP) is one of the most frequently used materials today.[2,3]

However, the bond with Y-TZP is difficult to establish due to its acid-resistant and silica-free surface.[4] Hydrofluoric acid etching and silanization techniques have been unsuccessful for Y-TZP bonding and are only effective for glass ceramic materials.[5,6] Successful cementation of Y-TZP ceramic is crucial for the clinical success of the restoration, as is the case with any restorative material.[7,8]

For this reason, some manufacturers and researchers have attempted to modify the surface properties of Y-TZP using different methods. Tribochemical silica coating has been developed for zirconia bonding to increase the extent of micromechanical interlocking and chemical adhesion.[2,9,10] There is no conclusive evidence as to whether the silica becomes permanently attached to the Y-TZP.[4] It can, however, be predicted that forceful water rinsing will be able to remove temporarily attached silica; whereas, chemically bonded silica would be retained.[4] The introduction of surface roughening by airborne-particle abrasion has been suggested as an alternative method for high-strength ceramics.[11] Nevertheless, it is important to note that air-abrasion treatment introduces microcracks, which may be a significant degrading factor in terms of the long-term performance of Y-TZP restorations.[12,13]

If mechanical damage is to be avoided, alternative techniques to create a bond between the zirconia and the luting system would need to rely on a chemical interaction.[14] Various zirconia primers have been developed in an attempt to attain chemically improved adhesion between the resin cement and the dental Y-TZP.[3] These are easy to apply and do not require expensive and complicated apparatuses. However, controversial reports have emerged concerning their hydrolytic stability and have shown that zirconia primers are not always a favorable option.[2]

Because the technique of using multi-step resin cement is time consuming and complicated, self-adhesive resin cements have recently been developed. Despite increases in the clinical use of self-adhesive resin cements, further evidence regarding the adhesive cementation of Y-TZP restorations is necessary to establish the most reliable technique.[15] However, there is a lack of data on the shear strength of bonding to zirconia ceramics with the newly developed zirconia primers and self-adhesive resin cements.

Therefore, the aim of this study is to evaluate the effect of zirconia primers, air-abrasion, and tribochemical surface treatment methods on the shear bond strength between Y-TZP ceramic and self-adhesive resin cement. The null hypotheses tested suggested that different surface treatment applications do not influence the strength of the bonding to Y-TZP ceramic.

METHODS AND MATERIALS

Preparation of the Specimens

Partially sintered zirconia blocks of 97% zirconium dioxide (ZrO_2) stabilized with 3% yttria (Lava Frame,

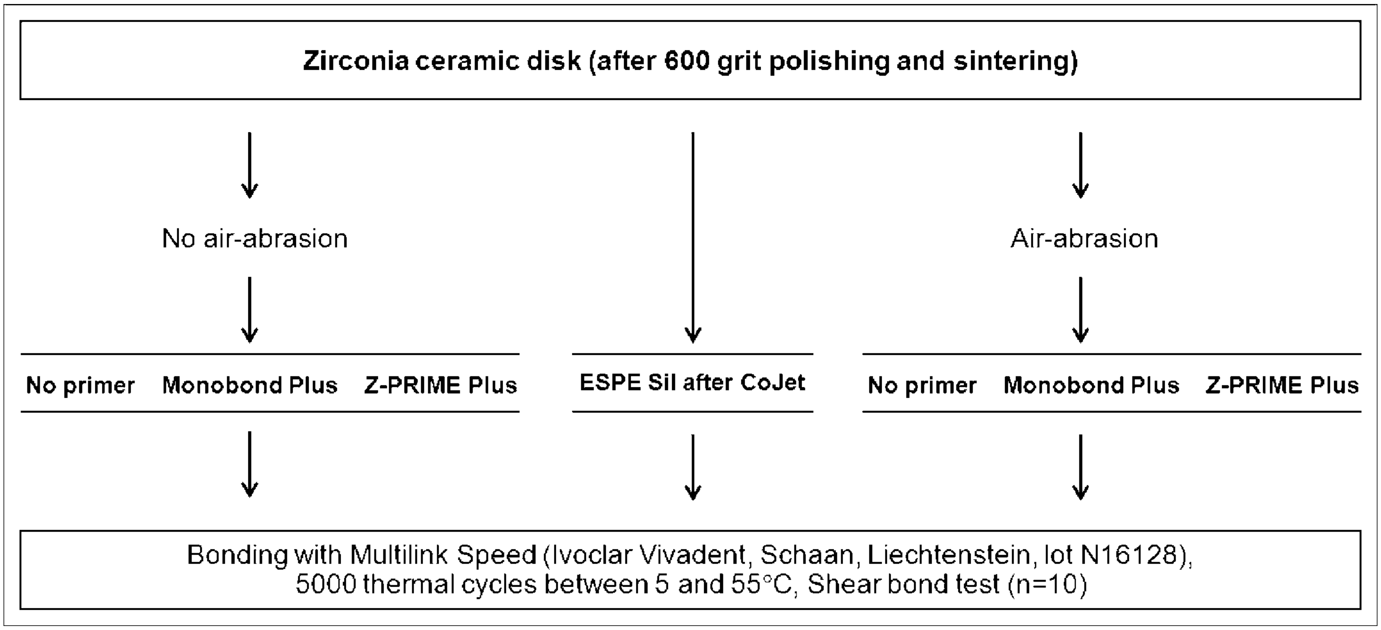

Figure 1. *Experimental design of the study.*

3M ESPE, St Paul, MN, USA), which were 19-mm in diameter and 100-mm high, were sectioned with a low-concentration diamond blade (Allied High Tech Productions Inc, Compton, CA, USA) to obtain 4-mm thick slices. The surfaces of each specimen were polished and ground with 600-grit silicon carbide abrasive using an underwater cooling method. The disk-shaped zirconia ceramics were ultrasonically cleaned in distilled water for three minutes, and then they were fully sintered using the manufacturer's instructions. Thereafter, they were embedded in polyethylene molds (19-mm inner diameter, 21-mm outer diameter, 12-mm height), with one side of the disk exposed for cement bonding.

Surface Treatments and Bonding Procedure

Subsequently, 70 specimens were randomly divided into seven groups according to the surface treatments and resin cement to be applied (N=70; n=10 per group). Figure 1 and Table 1 show the experimental design and the materials used in this study, respectively. Three of the groups were not subjected to air-abrasion and were directly treated with Monobond Plus (Ivoclar Vivadent, Schaan, Liechtenstein), Z-PRIME Plus (Bisco, Schaumburg, IL, USA), or did not undergo any primer treatment. Another three groups were subjected to air-abrasion with 50-μm grain size aluminum oxide (Al_2O_3) particles. Air-abrasion was conducted at a standoff distance of 10 mm with 3.5 bars of pressure for 15 seconds. The substrate surface was then rinsed for 30 seconds and air-dried for 30 seconds. The surface was then either treated with Monobond Plus or Z-PRIME Plus or not treated with any primer. The final group was subjected to tribochemical silica coating with the CoJet system (3M ESPE) using a 30-μm particle size. The substrate surface was then rinsed for 30 seconds and air-dried for 30 seconds.

Table 1: *Materials Used in the Study*

Brand Name	Composition	Batch Number	Manufacturer
CoJet	Silicatized sand (particle size, 30 μm) 30-42 psi 2-3 bar 15 seconds	368315	3M ESPE, St Paul, MN, USA
ESPE Sil	3-methacryloxyprophyltrimethoxy silane in ethanol	410841	3M ESPE, St Paul, MN, USA
Monobond Plus	Alcohol solution of silane methacrylate, phosphoric acid methacrylate and sulphide methacrylate	M35022	Ivoclar Vivadent, Schaan, Liechtenstein
Z-PRIME Plus	MDP, carboxlyate monomers	0900012783	BISCO, Schaumburg, IL, USA
Multilink Speed	Dimethacrylate, ytterbium trifluoride, adhesive monomer, MDP, glass, silicone dioxide	N16128	Ivoclar Vivadent, Schaan, Liechtenstein

Table 2: *Means and Standard Deviations for Shear Bond Strength Results (MPa) According to the Different Surface Treatments and Multilink Speed Cement*[a]

Priming Conditions	**Surface Conditions**[a]	
	No air-abrasion (polished)	Air-abrasion
None	3.91 (0.72)A	10.56 (3.32)B
Monobond-Plus	4.86 (1.77)A	8.93 (3.13)B
Z-PRIME plus	8.27 (2.79)B	16.50 (2.26)C
CoJet and ESPE Sil	8.54 (3.98)B	

[a] *Small caps that differ in each cell indicate statistical differences (p<0.05); whereas, identical small caps indicate no statistical difference in the designated group. Based on Student-Newman-Keuls multiple comparison test.*

The surface was then treated with a silane primer (ESPE Sil, 3M ESPE). Self-adhesive resin cement (Multilink Speed, Ivoclar Vivadent) was mixed according to the manufacturer's instructions and was placed inside a no. 5 size gel-cap (area=16.8 mm^2). The gel-cap was positioned on the zirconia surface and light-cured on four sides for 20 seconds per side using an LED light-curing unit (Eliper S10, 3M ESPE) at 600 mW/cm^2. For one hour at 23°C ± 1°C, the specimens were left to polymerize and then stored in distilled water for another 23 hours at 37°C. They were then subjected to 5000 thermal cycles between 5°C and 55°C. The dwelling time was 30 seconds at each temperature, and the transfer time between baths was two seconds. The pH values of the primers were measured by a pH electrode (Orion Glass Body ROSS Combination pH Electrode, Thermo Scientific, Beverly, MA, USA).

Bond Strength Test and Surface Analysis

The specimen was mounted in the jig of a universal testing machine (LF-Plus, Ametek Inc, Largo, FL, USA) and a load was applied to the adhesive interface (crosshead speed: 0.5 mm/min) until failure occurred. The corresponding software recorded the maximum stress (MPa) required to produce a failure. The failure mode was observed with a stereomicroscope (90×). An investigation of the resin bonding on the zirconia and fractured surfaces was carried out using a scanning electron microscope (SEM; S-4700 FESEM, Hitachi, Tokyo, Japan) at 150× and 3000× magnification and 10 kV accelerating voltage.

The formation of the monoclinic phase after air-abrasion and tribochemical surface treatments was determined with x-ray diffraction analysis (XRD; Diffractometer D5000, Siemens, Karlsruhe, Germany). The surfaces of the specimens were scanned with a copper Kα x-ray from 20° to 40° with a step size of 0.04° at five-second step intervals.

Statistical Analysis

The R programming language (R Foundation for Statistical Computing, Vienna, Austria)[16] was used to perform the data analysis. After checking the data normality and the equality of variance, a one-way analysis of variance (ANOVA) and a Student-Newman-Keuls multiple comparison test were performed. The mean difference was regarded as significant at the level of $p<0.05$.

RESULTS

The means and standard deviations for shear bond strength across all groups are presented in Table 2. A one-way ANOVA was used to calculate the statistical significance for the surface treatments ($p<0.05$). Air-abrasion, the use of a primer, and CoJet treatment were all more effective than the control group treatment, excluding the Monobond Plus treatment. The Z-PRIME Plus treatment after air-abrasion showed the best result.

The failure mode distribution for all samples is shown in Figure 2. The results showed a high percentage of adhesive failures at the zirconia surface in the control group. In contrast, when using additional surface treatments, the mixed failure mode was increased. All specimens presented mixed failure modes for the Z-PRIME Plus treatment group after air-abrasion.

Figures 3a-g show the representative SEM images (magnification, 150×) for Multilink Speed cement stubs on the contact area of the zirconia specimens. The remains of the resin cement can be seen to a relative degree (Figure 3b-g). It is notable that adhesive failure can be seen in the control group images (Figure 3a), yet the specimens treated with air-abrasion and Z-PRIME Plus exhibited mixed failure with resin cement and a characteristic corrugation appearance (Figure 3g).

Figure 4 shows the SEM images and energy dispersive spectroscopy (EDS) spectra for the surfaces conditioned with the CoJet system. A substantial number of silica particles could be seen on the zirconia surface after CoJet surface treatment. However, only a small amount of silica could be seen after the water rinsing, which indicates an air-abraded, roughened surface structure. The XRD results show the peak of the monoclinic phase in the case of sandblasting and the CoJet treatment groups compared with the Y-TZP control group

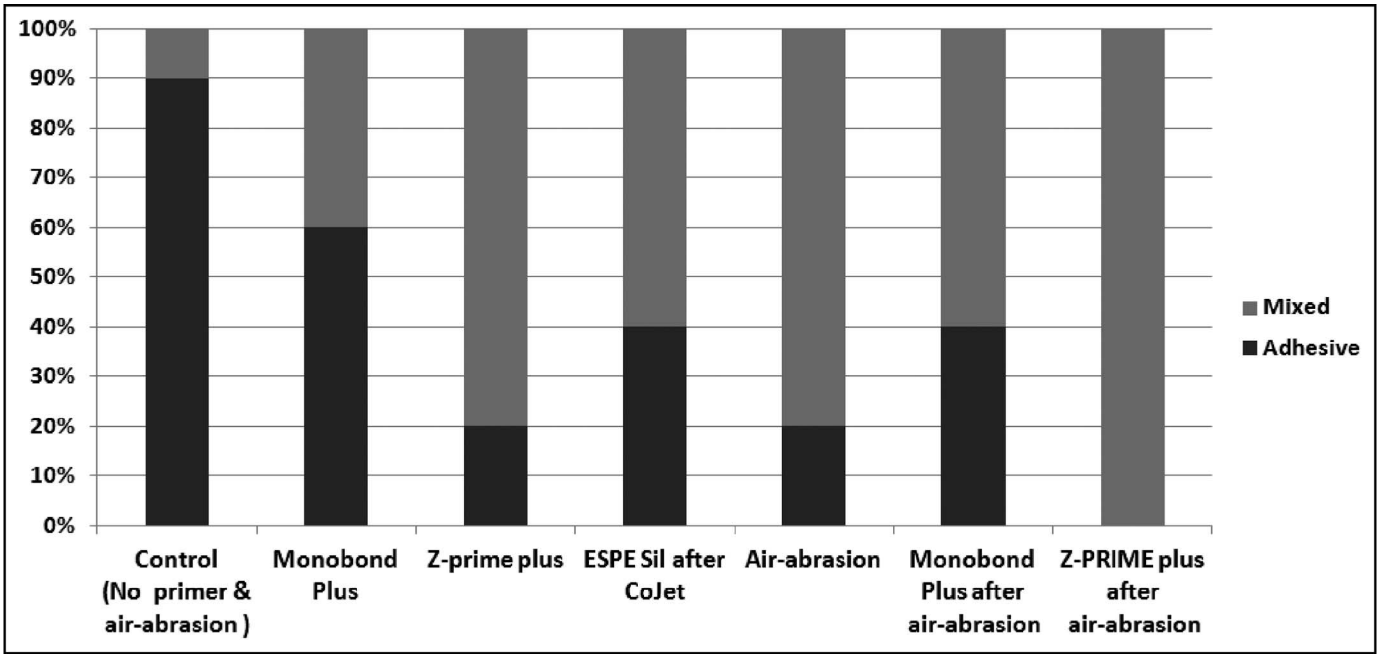

Figure 2. *Failure mode distribution.*

(Figure 5). The pH values of the primers were as follows, expressed in mean (SD): Monobond Plus, 1.63 (0.13); Z-PRIME Plus, 3.85 (0.04), and ESPE Sil, 4.39 (0.09).

DISCUSSION

This study investigated the effect of seven surface treatments on the shear bond strength of self-adhesive resin cement used on a Y-TZP ceramic. The results show that bonding was significantly affected by the air-abrasion, zirconia primer, and tribochemical silica coating treatments, and a combination of the air-abrasion method and the use of a zirconia primer showed the best result. Given that untreated zirconia surfaces presented the lowest bonding strength, the hypothesis of the study was nullified. This result is supported by previous studies that have shown that some conventional

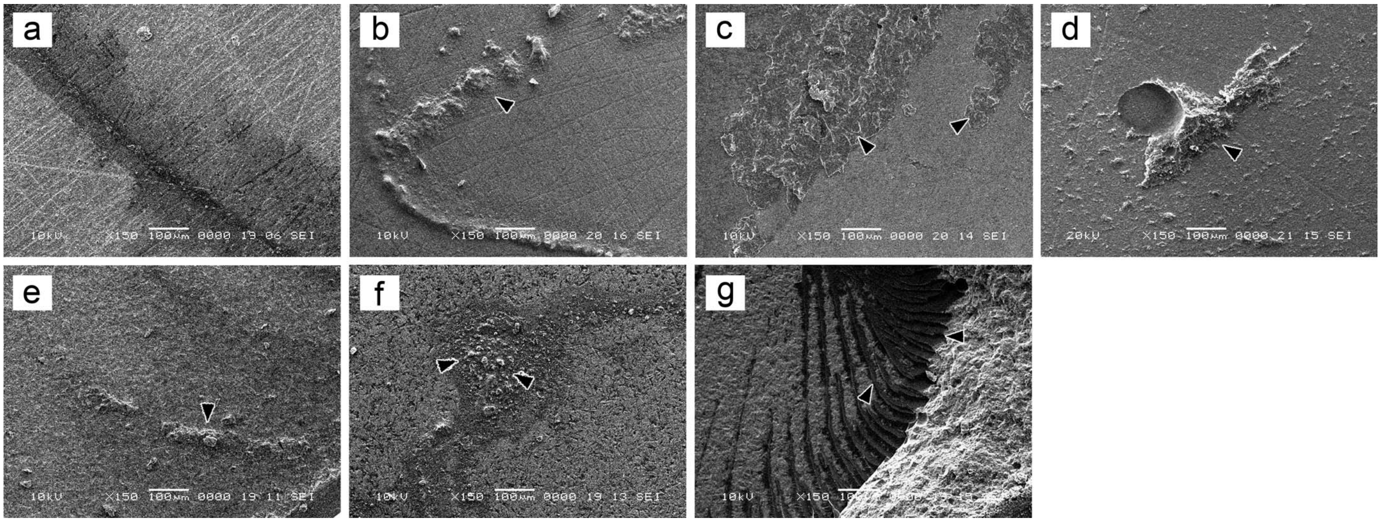

Figure 3. *Scanning electron micrographs (150× original magnification) of zirconia ceramic specimens cemented with Multilink Speed: (a) control surface without treatment; (b) surface coated with Monobond Plus; (c) surface coated with Z-PRIME Plus; (d) surface coated with ESPE Sil after tribochemical CoJet system treatment; (e) surface air-abrasion with 50 μm Al_2O_3; (f) surface coated with Monobond Plus after air-abrasion; and (g) surface coated with Z-PRIME Plus after air-abrasion. The arrows indicate the boundaries of the remaining resin cement.*

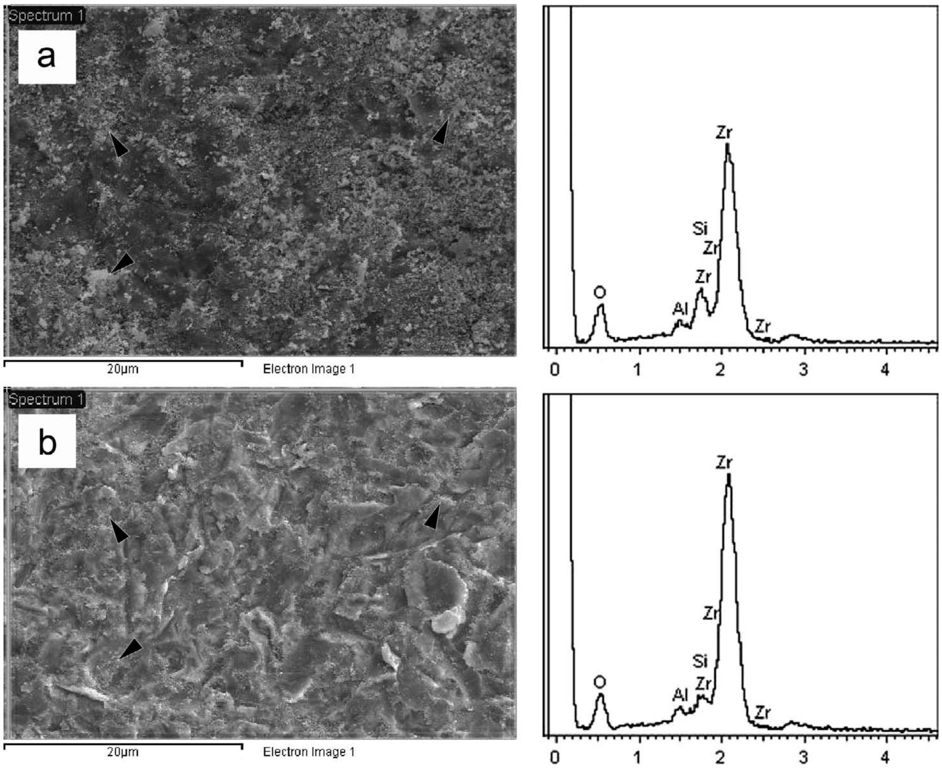

Figure 4. *SEM images (3000×) and EDS spectra: (a) surface conditioned with the tribochemical CoJet system; and (b) surface rinsed after tribochemical CoJet treatment. The small particles marked by the arrows indicate the remaining silica materials on the Y-TZP surface. The spectrum of the surface rinsed after the CoJet system treatment presents peaks of Al and Si. Zr, zirconium; Al, aluminum; O, oxygen; Si, silicon.*

and self-adhesive resin cements presented a low bonding strength when applied to an untreated zirconia ceramic surface.[17,18] These findings can be explained by the poor chemical interaction at the interfacial grain level between the hydroxyl groups of Y-TZP ceramics and the methacrylated phosphoric acid ester of self-adhesive resin cements such as Multilink Speed. Although this self-adhesive resin cement includes 10-methacryloyloxydecyldihydrogen phosphate (MDP), these phosphoric acid groups interacted chemically with ZrO_2,[17,19] and the functional monomer properties for the amount and flow appeared to be insufficient for increasing zirconia adhesion; the self-adhesive resin cement was insufficiently able to interact without any pretreatment.

The results showed that the bonding strength was significantly affected by 50-µm air-abrasion, regardless of whether the specimens were pretreated with zirconia primers. This result is in good agreement with those of previous studies.[17,18,20] The increase in roughness and surface energy that resulted from the air-abrasion method may have eased the resin cement flow into the microretention, thereby increasing the micromechanical interlocking between the resin cements and zirconia surfaces.[2,18,20] Moreover, an increased surface area on the Y-TZP would have been generated by air abrasion, increasing the chemical reaction with the phosphate groups of Multilink Speed as well as improving the micromechanical interlocking.

Two primers of different compositions were used in this study. Treating the Y-TZP surface with Z-PRIME Plus significantly increased the shear bonding strength for the intact zirconia surfaces and showed the best bonding strength result when combined with the air-abrasion pretreatment. Z-PRIME Plus contains two adhesive functional monomers (carboxylic and MDP monomers) that interact chemically with the zirconium oxide layer at the resin and zirconia interfaces. It is possible that these interfacial forces could have improved the surface wettability and chemical affinity of Y-TZP ceramics, thus increasing the interlocking with the self-adhesive resin cement. The synergic effect of the acidic MDP and carboxylate monomers is the most likely explanation for the higher bonding strength seen with this primer.[21] The bonding strength for Z-PRIME Plus surface treatment after air-abrasion was twice that when only the Z-PRIME Plus primer was applied. In this group, the failure mode results

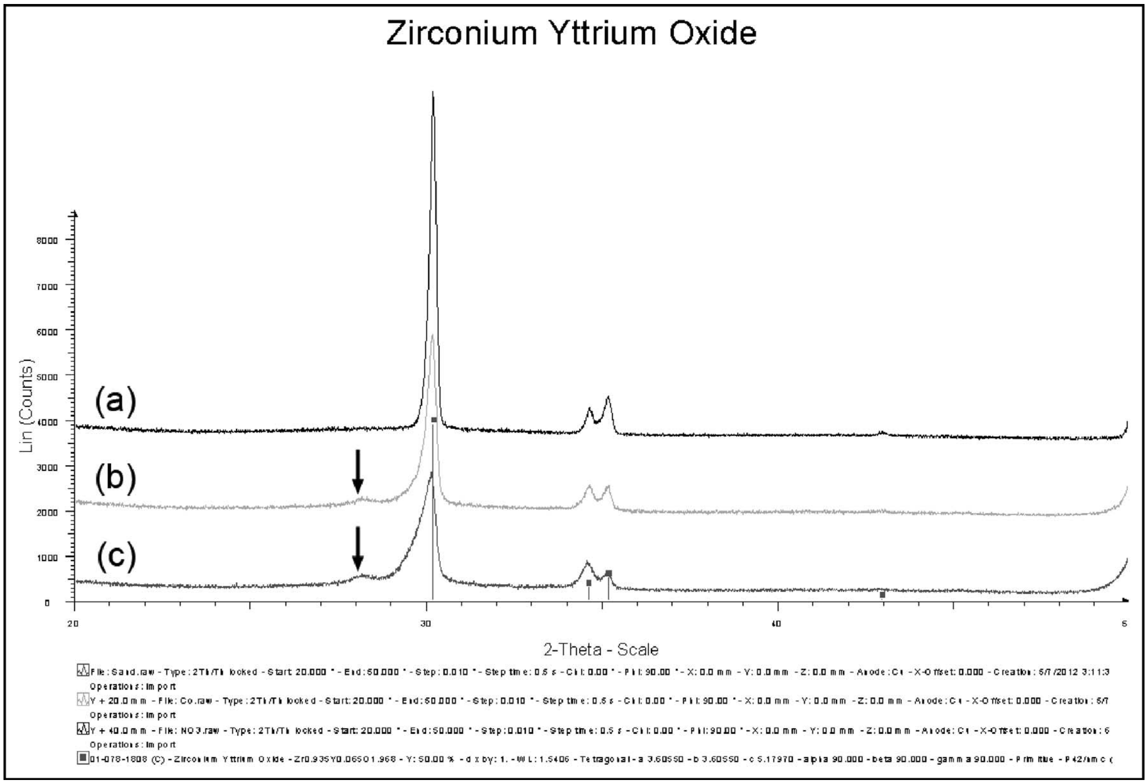

Figure 5. *XRD results after air-abrasion and CoJet treatment on zirconia surfaces: (a) control; (b) surface coated with 30 μm silica-modified Al_2O_3 particles; and (c) surface air-abrasion with 50 μm Al_2O_3. The arrows indicate monoclinic phase transformation.*

showed all mixed fracture patterns. This was due to a synergic effect produced by the increased contact area on the chemical interactions between the MDP and carboxylic monomers of the Z-PRIME Plus and the Y-TZP ceramic surface. Representative SEM images of this group and the air-abrasion group showed a unique corrugated fracture appearance, a mixed fracture pattern, and a thicker primer layer compared with the images for the samples treated with Monobond Plus primer. This corrugated appearance is considered to indicate the phenomenon of strong adhesion to resist the shear force. However, the Y-TZP surface treatment with the Monobond Plus primer did not significantly increase the shear bonding strength for the intact zirconia surfaces and showed adhesive and mixed failure modes. For the group with Monobond Plus application after air-abrasion pretreatment, the results showed that the bonding strength significantly increased compared with the control group, although this seems to have been a result of the air-abrasion rather than the primer. SEM images for the Monobond Plus group showed a thinner layer compared with those for the Z-PRIME Plus group and did not show a unique fracture appearance regardless of whether there was air-abrasion. Monobond Plus consists of silane methacrylate for glass ceramic, sulfide methacrylate for precious metal alloys, and phosphoric acid methacrylate for oxide ceramics and base metal alloys. Zirconia ceramic is composed of a glass-free material, which means that the silane cannot take effect. Monomers containing sulfur from 6-methacryloxyhexyl 2-thiouracil-5-carboxylate (MTU-6) and 6-[N- (4-vinylbenzyl) propylamino]-1,3,5-triazine 2,4-dithione (VBATDT) are purposely chosen to promote the adhesion to noble alloys; however, for bonding to base metal alloys, they are not always effective. Likewise, Monobond Plus has a low pH value of 1.63 because the phosphate monomer is acidic.[22,23] As such, some water and the silane component in acidic alcohol may be hydrolyzed. The silane priming ability may be reduced by polymerizing via condensation reaction to form the polysiloxane oligomer, and therefore there may be interference with zirconia bonding ability.[24,25]

Using the tribochemical CoJet system, the ceramic surface is abraded with silica-modified Al_2O_3 particles, leading to silica inclusions in the ceramic surfaces. A chemical bond between the organic resin materials and the silica-modified zirconia surfaces may be formed after silica-coating due to the silanization that occurs with a silane coupling agent.[26] However, there is a lack of consensus about whether the silica is chemically bonded to zirconia or merely weakly attached. The tribochemical modification mechanism where silica attaches to a densely sintered and mechanically tough zirconia surface is still not well understood.[21] However, achieving a stable and permanent attachment is generally perceived as extremely important in the wider aim of achieving strong and durable bonds to zirconia. In the present study, SEM, EDS, and a shear bond strength test were used to investigate whether the tribochemical coating/silane treatment was effective for improving zirconia bonding. Both the SEM and EDS data confirmed that the silica nanoparticles were easily removed simply by rinsing with a forceful water stream. ESPE Sil application after the water rinsing did not improve the bonding strength. Indeed, this bonding strength was only similar to the result for the air-abrasion group, which most likely explains why the 30-μm silica particle size increased the surface roughness of the zirconia surface.

Phase transformation of Y-TZP may be caused by air-abrasion, which will create microcracks. Unfavorable changes to the superior mechanical properties of the material may be caused.[27,28] Although the bonding strength was significantly affected by air-abrasion and tribochemical silica coating due to this method increasing the surface contact area, the representative phenomenon of phase transformation was observed in the XRD results. Zirconia is prone to low temperature degradation due to the metastability of its phase transformation from a tetragonal to a monoclinic phase. The long-term effects of phase transformation on zirconia ceramics still requires further research.

CONCLUSIONS

Within the limitations of the current experimental settings, it can be concluded that the application of self-adhesive resin cement without pretreatment was not sufficient for improving the strength of the bonding to an untreated zirconia ceramic surface. The combined use of the air-abrasion method and a zirconia primer appears to be a reliable method of achieving strong and durable bonding between zirconia ceramics and self-adhesive resin cements. It should also be noted that using air-abrasion with a 50-μm particle size and a CoJet application of 30-μm particle size can lead to a phase transformation on the Y-TZP surface.

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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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