

# Air Abrasion Before and/or After Zirconia Sintering: Surface Characterization, Flexural Strength, and Resin Cement Bond Strength

FO Abi-Rached • SB Martins • AA Almeida-Júnior  
GL Adabo • M Sousa Góes • RG Fonseca

## Clinical Relevance

The air abrasion performed before and after zirconia sintering can provide stronger bond strength at the zirconia–resin cement interface as well as an increase in the short-term flexural strength.

## SUMMARY

The purpose of this *in vitro* study was to evaluate the effect of air-abrasion/zirconia sintering order on the yttria partially stabilized tetragonal zirconia polycrystal (Y-TZP) surface characterization (roughness, morphology, and phase transformation), flexural strength (FS), and shear bond strength (SBS)

Filipe de Oliveira Abi-Rached, DDS, MSc, PhD, adjunct professor, Department of Dental Materials and Prosthodontics, Araraquara Dental School, Unesp – Univ Estadual Paulista, Araraquara, São Paulo, Brazil

Samira Branco Martins, DDS, MSc, PhD student, Department of Dental Materials and Prosthodontics, Araraquara Dental School, Unesp – Univ Estadual Paulista, Araraquara, São Paulo, Brazil

Antonio Alves de Almeida-Júnior, DDS, MSc, PhD, assistant professor, Tiradentes University – UNIT, Aracaju, Sergipe, Brazil

Gelson Luis Adabo, DDS, MSc, PhD, professor, Department of Dental Materials and Prosthodontics, Araraquara Dental School, Unesp – Univ Estadual Paulista, Araraquara, São Paulo, Brazil

to a resin cement. Y-TZP specimens were air abraded with 50- $\mu\text{m}$   $\text{Al}_2\text{O}_3$  particles after (AS), before (BS), or before and after zirconia sintering (BAS). For roughness (Ra), 30 block specimens (12×12×3.0 mm; n=10) had their surfaces analyzed by a profilometer. Next, on the air-abraded surfaces of these specimens, composite resin discs (n=30) were bonded with RelyX ARC. The bonded specimens were stored for 24 hours in distilled water at 37°C before shear testing. Failure mode was determined

Marcio Sousa Góes, Lic Chem, MSc, PhD, associate professor, Federal University of Latin American Integration – UNILA, Foz do Iguaçu, Paraná, Brazil

\*Renata Garcia Fonseca, DDS, MSc, PhD, associate professor, Department of Dental Materials and Prosthodontics, Araraquara Dental School, Unesp – Univ Estadual Paulista, Araraquara, São Paulo, Brazil

\*Corresponding author: Rua Humaitá, no. 1680 – 4° andar, Araraquara, São Paulo, Brazil, 14801-903; e-mail: renata@foar.unesp.br

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with a stereomicroscope (20×). The surface morphology ( $n=2$ ) was evaluated by SEM (500×). For the four-point flexural strength test (EMIC DL2000), 39 bar-shaped specimens ( $20 \times 4.0 \times 1.2$  mm;  $n=13$ ) were air abraded according to the three conditions proposed, and an additional group (nonabraded) was evaluated ( $n=13$ ). The quantitative analysis of phase transformation ( $n=1$ ) was completed with Rietveld refinement with X-ray diffraction data. Ra ( $\mu\text{m}$ ) and SBS (MPa) data were analyzed by one-way analysis of variance (ANOVA) and the Tukey test ( $\alpha=0.05$ ). Pearson correlation analysis was used to determine if there was a correlation between roughness and SBS. For FS (MPa) data, one-way ANOVA and the Dunnett C-test ( $\alpha=0.05$ ) were used. The air-abrasion/zirconia sintering order influenced significantly ( $p<0.001$ ) Ra, SBS, and FS. The BS and AS groups presented the highest (1.3  $\mu\text{m}$ ) and the lowest (0.7  $\mu\text{m}$ ) Ra. The highest SBS (7.0 MPa) was exhibited by the BAS group, followed by the AS group (5.4 MPa) and finally by the BS group (2.6 MPa). All groups presented 100% adhesive failure. A weak correlation ( $r=-0.45$ ,  $p<0.05$ ) was found between roughness and SBS. The air-abrasion/zirconia sintering order provided differences in the surface morphology. The nonabraded (926.8 MPa) and BS (816.3 MPa) groups exhibited statistically similar FS values but lower values than the AS (1249.1 MPa) and BAS (1181.4 MPa) groups, with no significant difference between them. The nonabraded, AS, BS, and BAS groups exhibited, respectively, percentages of monoclinic phase of 0.0 wt%, 12.2 wt%, 0.0 wt%, and 8.6 wt%. The rougher surface provided by the air-abrasion before zirconia sintering may have impaired the bonding with the resin cement. The morphological patterns were consistent with the surface roughness. Considering the short-term SBS and FS, the BAS group exhibited the best performance. Air abrasion, regardless of its performance order, provides tetragonal to monoclinic transformation, while sintering tends to zero the monoclinic phase content.

## INTRODUCTION

Yttria partially stabilized tetragonal zirconia polycrystal (Y-TZP) has been widely used to manufacture metal-free fixed partial dentures or implant-supported prostheses due to its optical properties,<sup>1</sup>

biocompatibility,<sup>2</sup> low thermal conductivity,<sup>3</sup> chemical stability,<sup>4</sup> as well as its high fracture toughness and mechanical performance when compared to the other dental ceramics.<sup>5</sup> In minimally retentive situations, the resin cements might be a good option<sup>6</sup> because of their improved mechanical properties when compared to zinc phosphate and glass ionomer cements<sup>7</sup> and also because of the possible chemical interactions between zirconia surface and the resin cement components (adhesive cementation).<sup>8,9</sup> With regard to micromechanical retention, which contributes significantly to improve the bonding between zirconia and resin cements,<sup>10-13</sup> although there are different methods to roughen zirconia surface, such as nanostructured alumina coating,<sup>14</sup> laser,<sup>15,16</sup> selective infiltration-etching (SIE),<sup>17,18</sup> and hot-etching solution,<sup>18</sup> among others, air abrasion with alumina ( $\text{Al}_2\text{O}_3$ ) particles is still an effective and one of the most applicable methods.<sup>10,15,16,18</sup>

Air abrasion can be performed with  $\text{Al}_2\text{O}_3$  particles of different sizes and is usually carried out after zirconia sintering and prior to cementation. However, since zirconia is a densely sintered material and consequently exhibits high hardness,<sup>5</sup> it is difficult to roughen its surface,<sup>19</sup> requiring higher air pressure and/or coarser  $\text{Al}_2\text{O}_3$  particles capable of promoting a desirable surface roughness. On the other hand, if this procedure is severe, it may create surface flaws that can propagate into the bulk of the zirconia, compromising its mechanical properties.<sup>20,21</sup> Another way to solve this question would be performing air abrasion before the zirconia sintering, that is, when this material does not exhibit such high hardness. This simple modification may allow the use of smaller particles to provide a surface whose roughness and morphology are favorable to the adhesive bonding at the zirconia-cement interface without jeopardizing the mechanical strength of the zirconia. Monaco and others<sup>22</sup> observed that regardless of the particle size evaluated (30, 50, and 110  $\mu\text{m}$ ), the air abrasion performed before zirconia sintering provided higher roughness in comparison with that performed after sintering. However, Monaco and others<sup>23</sup> and Moon and others<sup>24</sup> reported no significant differences in the shear bond strength between the groups abraded before and after zirconia sintering. Besides the increase in zirconia roughness reported by Monaco and others,<sup>22</sup> another very important aspect observed by these authors, as well as by Moon and others,<sup>24</sup> was the decrease of the monoclinic phase when abrasion was performed before zirconia sintering.

Besides the few studies<sup>22,24</sup> that have investigated the effect of the air abrasion performed before and after zirconia sintering on its roughness and adhesive bonding, there is no consensus with respect to the influence of the air-abrasion/zirconia sintering order on roughness. Moreover, the association between the air abrasion performed before and after zirconia sintering is another viable option to be investigated. In addition, it would be important to evaluate the influence of the air-abrasion/zirconia sintering order not only on phase transformation but also on the mechanical strength of the zirconia.

Thus, the purpose of this *in vitro* study was to evaluate the effect of the air-abrasion/zirconia sintering order (air abrasion performed after, before, or before and after zirconia sintering) on the Y-TZP ceramic surface characterization (roughness, morphology, and phase transformation) and flexural strength (FS) and also its efficacy on the shear bond strength (SBS) at the zirconia–resin cement interface. The null hypothesis was that the air-abrasion/zirconia sintering order does not modify zirconia roughness, its flexural strength, or its bond strength with a resin cement.

## METHODS AND MATERIALS

### Preparation of Zirconia Specimens

Thirty block specimens (15×15×3.5 mm) were prepared for roughness analysis and SBS test, while 52 bar-shaped specimens (25×5.0×1.5 mm) were prepared for four-point flexural strength testing (ISO 6872).<sup>25</sup> The specimens were obtained by cutting presintered zirconia frames (Lava, 3M ESPE AG, Seefeld, Germany) with a sectioning machine (Iso-met 1000, Buehler Ltd, Lake Bluff, IL, USA) using a diamond-coated disc saw (Diamond Wafering Blade, Series 15LC no. 11-4276, Buehler) under water irrigation. The specimens were washed in tap water to remove the cutting debris, and their ends were finished manually using a ceramic polisher (Exa Cerapol 0361HP, Edenta AG, Au, SG, Switzerland) in a low-speed hand piece.

The specimens were air abraded with 50- $\mu$ m Al<sub>2</sub>O<sub>3</sub> particles (Bio-Art Equip. Odontol. Ltda, São Carlos, SP, Brazil) after (AS), before (BS), or before and after zirconia sintering (BAS). For the four-point flexural strength test, 39 specimens were obtained according to the three air-abrasion conditions proposed, and an additional group (nonabraded) was included (n=13).

The sintering process was performed in a specific oven (Lava Furnace 200, Dekema Dental-Keramiköfen GmbH, Freilassing, Germany) according to the

manufacturer's instructions (heating rate=20°C/min: 0°C-1000°C; 10°C/min: 1000°C-1500 °C; holding time=2 h and cooling rate=15°C/min: 1500°C-800°C; 20°C/min: 800°C-250°C; the oven was opened at 250°C). The dimensions of the specimens after sintering were 12×12×3.0 mm for roughness and SBS and 20×4.0×1.2 mm for flexural strength. For the air-abrasion procedure, the specimens were mounted on a holder (developed for each specimen shape) at a 90-degree angle and a distance of 10 mm from the tip of the air-abrasion unit (Basic Classic, Renfert GmbH, Hilzingen, Germany).<sup>26,27</sup> The specimens were air abraded for 20 seconds and 15 seconds at a pressure of 0.05 and 0.28 MPa for abrasion before and after sintering, respectively. The parameters (pressure and time) used for air abrasion before zirconia sintering were determined after some preliminary experiments. After sintering, all specimens were cleaned in 99% isopropanol using an ultrasonic cleaner for 10 minutes and left to dry at room temperature for 24 hours. Both analyses (surface roughness and SBS) were performed on the same specimens of each group.

### Surface Roughness Measurements

The surface roughness of all specimens was determined after their sintering, using a profilometer (SurfTest SJ-400, Mitutoyo Corporation, Kawasaki-shi, Japan) with a cutoff value ( $\lambda_c$ ) of 0.8 mm.<sup>16</sup> A diamond stylus with a 5- $\mu$ m tip radius at 0.5 mm/s and resolution of 0.01 mm examined a surface length of 4.0 mm. Three equidistant parallel measurements were made perpendicularly to the direction of the air abrasion with the stylus at a 90-degree angle on different areas of the specimen. The average reading was designated as the Ra ( $\mu$ m) value of each specimen evaluated. A single calibrated operator (intraclass correlation coefficient=0.89) recorded all measurements.

### Bonding Procedure and SBS Test

Thirty composite resin discs (Z100, 3M ESPE, St Paul, MN, USA) were produced using a custom-made metal split matrix (4.0-mm internal diameter and 2.0-mm thickness) positioned between two glass slabs covered with transparent polyester films. The light curing (Radii-Cal light-curing unit, SDI Ltd, Bayswater, Australia) was performed for 40 seconds on the top surface and two diametrically opposed sides of the resin discs (total of 120 s) at a light intensity of 800 mW/cm<sup>2</sup>. After the metal matrix was removed, the sides were light cured, taking care not to polymerize the bottom surface of the resin discs.

Table 1: Scheme of XRD Measurements

| Nonabraded             | AS                     | BS                      | BAS                                    |
|------------------------|------------------------|-------------------------|--|
| Nonabraded/nonsintered | Nonabraded/sintered    | Air abraded/nonsintered | Air abraded 1/nonsintered              |
| First measurement      | First measurement      | First measurement       | First measurement                      |
| Sintering process      | Air-abrasion procedure | sintering process       | Sintering process                      |
| Second measurement     | Second measurement     | Second measurement      | Second measurement                     |
| —                      | —                      | —                       | Air-abrasion procedure (air abraded 2) |
| —                      | —                      | —                       | Third measurement                      |

RelyX ARC resin cement (Bis-GMA, TEGDMA, silanated zirconia/silica filler, 3M ESPE) was proportioned by weight (0.010 g of each paste) and mixed for 10 seconds, and the composite resin discs were immediately bonded to the air-abraded zirconia surfaces. Next, a load of 1000 g was applied on top of the composite resin disc for five minutes.<sup>28</sup> After excess removal, the cement was light cured in two different positions (equidistant sides) for 40 seconds each.

The composite resin disc was inserted in a metal matrix (25-mm diameter) with a circular opening (4.2-mm diameter) with the zirconia block upward. Polyvinyl chloride tubes (20 mm in diameter and 20 mm high) were centrally positioned over the matrix and filled with polymethyl methacrylate (PMMA) autopolymerizing acrylic resin (Jet, Classico Odontological Goods Ltd, São Paulo, SP, Brazil), assembling the air-abraded zirconia surface to remain exactly at the same level of PMMA resin. All specimens were stored for 24 hours in distilled water at 37°C.

Each specimen was mounted on a metal holder in a mechanical testing machine (model DL2000, EMIC Equipment and Systems Testing Ltd, São José dos Pinhais, PR, Brazil), and a uniaxial compressive force was applied at the cement–zirconia interface by means of a knife-edged blade running at a crosshead speed of 0.5 mm/min until failure. SBS values were recorded in MPa.

### Failure Analysis

Debonded specimens were examined under a stereomicroscope (model M80, Leica Microsystems Ltd, Heerbrugg, Switzerland) at 20× magnification by a single trained observer, and the failure mode was classified as adhesive (complete zirconia surface was visible), cohesive within the cement layer or within the composite resin (almost all of the fracture surface was covered with cement or with composite resin), or mixed (a combination of adhesive and

cohesive), according to the dominant mode of failure in each quadrant of the zirconia surface.<sup>29</sup>

### Surface Morphology Analysis

For the surface morphology analysis, two additional specimens from each experimental group were mounted on metallic stubs and analyzed under a field emission scanning electron microscope (model JSM-7500F, JEOL Ltd, Peabody, MA, USA), which operated at 500× magnification with an accelerating voltage of 2.0 kV.

### Four-Point Flexural Strength Test

For the four-point flexural strength test (ISO Standard 6872)<sup>25</sup>, the specimens were positioned over two 0.8-mm-radius rounded bearers with a span distance of 16 mm. Two rounded loading pistons (0.8-mm radius, distance of 8 mm) running at a crosshead speed of 1.0 mm/min applied a uniaxial compressive force to the nonabraded surface, while for the air-abraded groups, the treated surface was submitted to the tensile load until failure. The test was performed at room temperature in a mechanical testing machine (model DL2000, EMIC Equipment and Systems Testing). The flexural strength values (MPa) were calculated according to the equation recommended by the ISO standard.

### X-ray Diffraction Analysis

The X-ray diffraction (XRD) analysis assessed the effect of the air-abrasion/zirconia sintering order on the phase transformation of zirconia. Table 1 presents the scheme of XRD measurements according to the experimental groups.

The XRD data (n=1) were collected using a RIGAKU RINT2000 rotating anode diffractometer (40 kV, 70 mA) with Cu  $\alpha$  radiation ( $\lambda_{\alpha 1}=1.5405$  Å,  $\lambda_{\alpha 2}=1.5443$  Å,  $I_{\alpha 1}/I_{\alpha 2}=0.5$ ) monochromatized by a curved graphite crystal. An interval from 20° to 120° (2 $\theta$ ) with a step size of 0.02° (2 $\theta$ ), 4 seconds per step, divergence 0.5, and open receiving slits, were the

| Table 2: Mean ( $\pm$ SD) of Ra ( $\mu$ m), SBS (MPa), and FS (MPa) Values <sup>a</sup> |                            |                            |                                 |
|---|----------------------------|----------------------------|---------------------------------|
|   | Ra                         | SBS                        | FS                              |
| non-abraded   | -                          | -                          | 926.8 $\pm$ 95.4 <sup>b</sup>   |
| AS  | 0.7 $\pm$ 0.1 <sup>c</sup> | 5.4 $\pm$ 0.6 <sup>b</sup> | 1249.1 $\pm$ 303.9 <sup>a</sup> |
| BS  | 1.3 $\pm$ 0.1 <sup>a</sup> | 2.6 $\pm$ 0.9 <sup>c</sup> | 816.3 $\pm$ 112.4 <sup>b</sup>  |
| BAS   | 1.0 $\pm$ 0.1 <sup>b</sup> | 7.0 $\pm$ 1.1 <sup>a</sup> | 1181.4 $\pm$ 262.7 <sup>a</sup> |

<sup>a</sup> Different letters indicate significant differences in columns ( $p < 0.05$ ).

selected conditions for Rietveld refinement.<sup>30</sup> The Rietveld refinements were performed using the General Structure Analysis System program<sup>31</sup> suite with EXPGUI interface.<sup>32</sup> The peak profile function was modeled using a convolution of the Thompson-Cox-Hastings pseudo-Voigt function (pV-TCH),<sup>33</sup> using the asymmetry function described by Finger and others,<sup>34</sup> which accounts for the asymmetry resulting from axial divergence. The bidimensional model for crystallite size described by Larson and Von Dreele<sup>31</sup> was used to account for the anisotropy in the half width of the reflections, and the model described by Stephens.<sup>35</sup> The following parameters were refined: atomic coordinates, occupancies, unit cell, scale factor, sample displacement, atomic displacement, and full width at half maximum. The crystal structure parameter used as basis of the Inorganic Crystal Structure Database code was 66781 (ZrO<sub>2</sub>, tetragonal), 18190 (ZrO<sub>2</sub>, monoclinic), and 53998 (ZrO<sub>2</sub>, cubic).

Statistical Analysis

The Shapiro-Wilk test indicated that the normality assumption for all data was satisfied, while the homogeneity by Levene test proved to be violated ( $p=0.001$ ) only for FS (MPa) data. Surface roughness ( $\mu$ m) and SBS (MPa) data were analyzed by one-way analysis of variance (ANOVA) followed by the Tukey honestly significant difference (HSD) *post hoc* test ( $\alpha=0.05$ ) to determine differences among the means. In addition, to test for a possible correlation between roughness and SBS, a linear correlation  $r$  was calculated by Pearson correlation analysis. The analysis of FS (MPa) data was performed by one-way ANOVA and the Dunnett C-test ( $\alpha=0.05$ ). Statistical analysis was performed using IBM SPSS Statistics (version 20, IBM Corp, Armonk, NY, USA).

RESULTS

According to the results of the one-way ANOVA, the air-abrasion/zirconia sintering order significantly influenced surface roughness ( $F=70.1$ ,  $p<0.001$ ), SBS ( $F=65.4$ ,  $p<0.001$ ), and FS ( $F=12.0$ ,  $p<0.001$ ). Table 2 shows the mean Ra ( $\mu$ m) and SBS (MPa) values, standard deviations, and statistical analysis

results identified with the Tukey HSD test and the FS (MPa) mean values, standard deviations, and statistical results obtained by the Dunnett C-test. The BS group presented the highest Ra value ( $\mu$ m), while the AS group yielded the lowest. The highest SBS value was exhibited by the BAS group, followed by the AS group and finally by the BS group. The failure mode observed was 100% adhesive in all groups. A weak correlation ( $r=-0.45$ ,  $p<0.05$ ) was found between roughness and SBS. The nonabraded and BS groups exhibited statistically similar FS values but lower values than the AS and BAS groups, with no significant differences between them.

The representative SEM images (Figure 1) indicated that the groups abraded before zirconia sintering (BS and BAS groups) exhibited more prominent microretentive grooves when compared to the smoother surface of the AS group. The BAS group exhibited a surface texture similar to that presented by the BS group but with more rounded edges.

Table 3 lists the results of quantitative phase analysis, and Figure 2 presents the representative diffraction patterns of the experimental groups according to each step performed to obtain them. Air abrasion provided an increase in the monoclinic phase for the BS and BAS/air-abraded 1 groups and a “decomposition” of t-ZrO<sub>2</sub> and c-ZrO<sub>2</sub> phases in others (t-ZrO<sub>2</sub> and m-ZrO<sub>2</sub>) for the AS and BAS/air-abraded 2 groups. The sintering process promoted the total incorporation of monoclinic phase into tetragonal and/or cubic phases.

DISCUSSION

The null hypothesis of the present study was rejected since the air-abrasion/zirconia sintering order influenced roughness, shear bond strength, and flexural strength. The air abrasion performed before zirconia sintering (BS group) provided the roughest surface, followed by the BAS and AS groups. The higher roughness provided by the air abrasion with 50- $\mu$ m Al<sub>2</sub>O<sub>3</sub> particles before sintering in comparison with that performed after sintering was also observed by

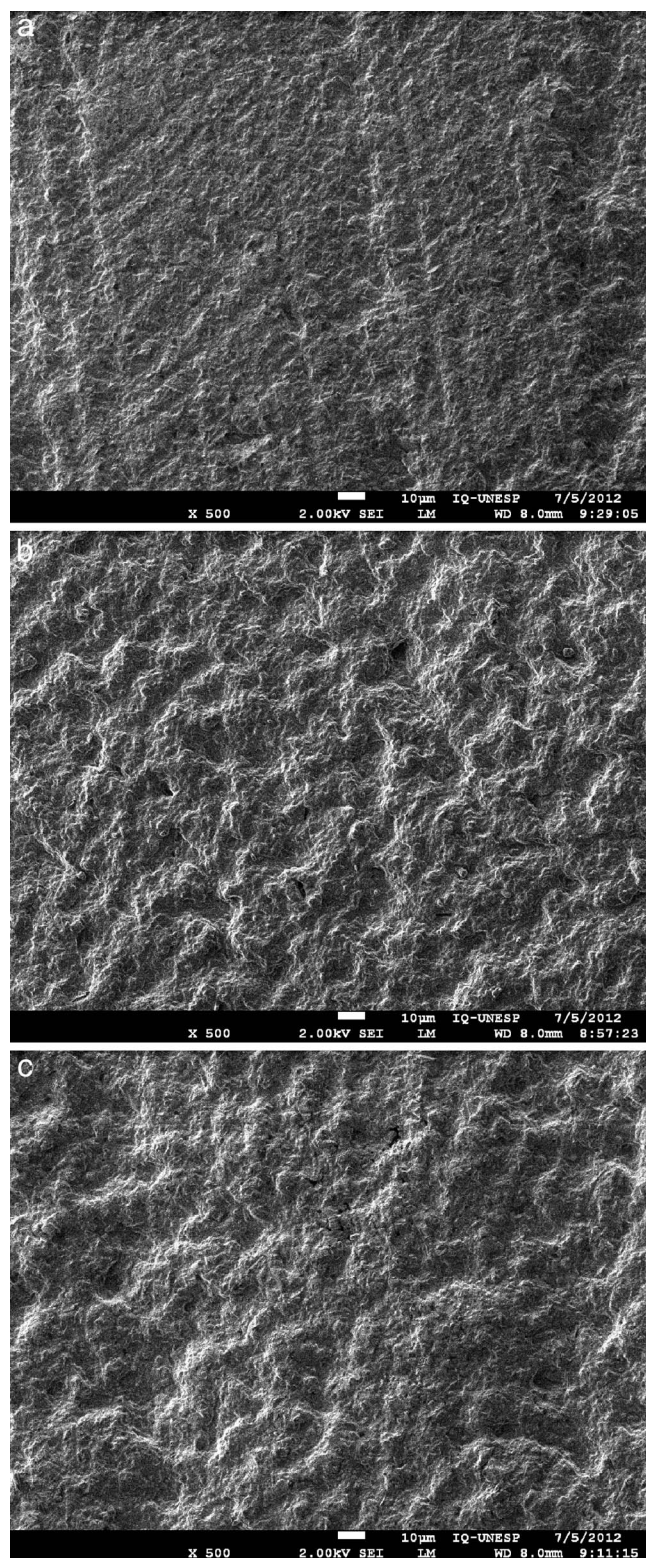


Figure 1. SEM images (500×) of the experimental groups according to the air-abrasion/zirconia sintering order. (a): After zirconia sintering (AS). (b): Before zirconia sintering (BS). (c): Before and after zirconia sintering (BAS).

Monaco and others<sup>22</sup> and may be due to the lower hardness of the zirconia in its green stage, which favors the imprint of its surface by the harder  $\text{Al}_2\text{O}_3$  particles. Regarding the morphological pattern, which was in line with roughness, we observed that the BS group exhibited more prominent microretentive grooves when compared to the smoother surface of the AS group, as reported by Monaco and others.<sup>22</sup> The BAS group, which was not evaluated by these authors,<sup>22</sup> exhibited a surface texture similar to that presented by the BS group but with more rounded edges, probably resulting from the air abrasion performed after sintering. Considering that the effect of the air-abrasion/zirconia sintering order has been poorly investigated, no additional information was found to further discuss our results.

Regarding the shear bond strength, the lowest mean value observed for the BS group probably is related to its higher roughness accompanied by the prominent edges observed by SEM, which probably impaired the wettability of the zirconia by the resin cement, considering that in this study a bonding agent was not used. On the other hand, the lowest roughness and a “flatter” morphology exhibited by the AS group may have been unfavorable to the micromechanical retention at the zirconia–resin cement interface, explaining its intermediate SBS value. The highest SBS mean value of the BAS group probably resulted from its intermediate roughness and morphology, which allowed a higher wettability of the zirconia by the resin cement when compared to the BS group and a higher micromechanical retention when compared with the AS group. Contrary to our SBS results, some studies<sup>23,24,26</sup> reported statistical similarity between the groups abraded before (BS group) and after (AS group) zirconia sintering. The difference in behavior between those studies<sup>23,24,26</sup> and ours may be related to the use or nonuse of a bonding agent.

The weak correlation between roughness and bond strength observed in this study can be corroborated by the findings of Winkler and Moore.<sup>36</sup> These authors evaluated the correlation between these two properties, varying the direction of the roughness measurements, that is, parallel or perpendicular to the scratches, and they concluded that when the reading was parallel, a correlation was observed. On the other hand, when the reading was perpendicular, as performed in the present study, the correlation was significantly lower. Also, in the study by Subaşı and Inan,<sup>16</sup> no significant correlation was observed when the relationships between roughness and bond strength values were compared



| Table 3: Phase Content (wt%) of the Experimental Groups |     |             |          |            |             |             |          |               |          |               |
|---|-----|-------------|----------|------------|-------------|-------------|----------|---------------|----------|---------------|
| Phases  |     | Nonabraded  |          | AS         |             | BS          |          | BAS           |          |               |
|   |     | Nonsintered | Sintered | Nonabraded | Air abraded | Air abraded | Sintered | Air abraded 1 | Sintered | Air abraded 2 |
| t-ZrO <sub>2</sub>                                      | wt% | 85.5(1)     | 89.2(1)  | 89.2(1)    | 59.2(5)     | 83.7(7)     | 74.6(2)  | 83.5(7)       | 73.2(3)  | 51.0(4)       |
| m-ZrO <sub>2</sub>                                      |     | 14.4(1)     | —        | —          | 12.2(6)     | 16.3(3)     | —        | 16.5(3)       | —        | 8.6(2)        |
| t-ZrO <sub>2</sub>                                      |     | —           | —        | —          | 28.6(1)     | —           | —        | —             | —        | 40.3(5)       |
| c-ZrO <sub>2</sub>                                      |     | —           | 10.7(1)  | 10.7(1)    | —           | —           | 25.4(7)  | —             | 26.8(8)  | —             |

Abbreviations: t, tetragonal ; m, monoclinic ; c, cubic.

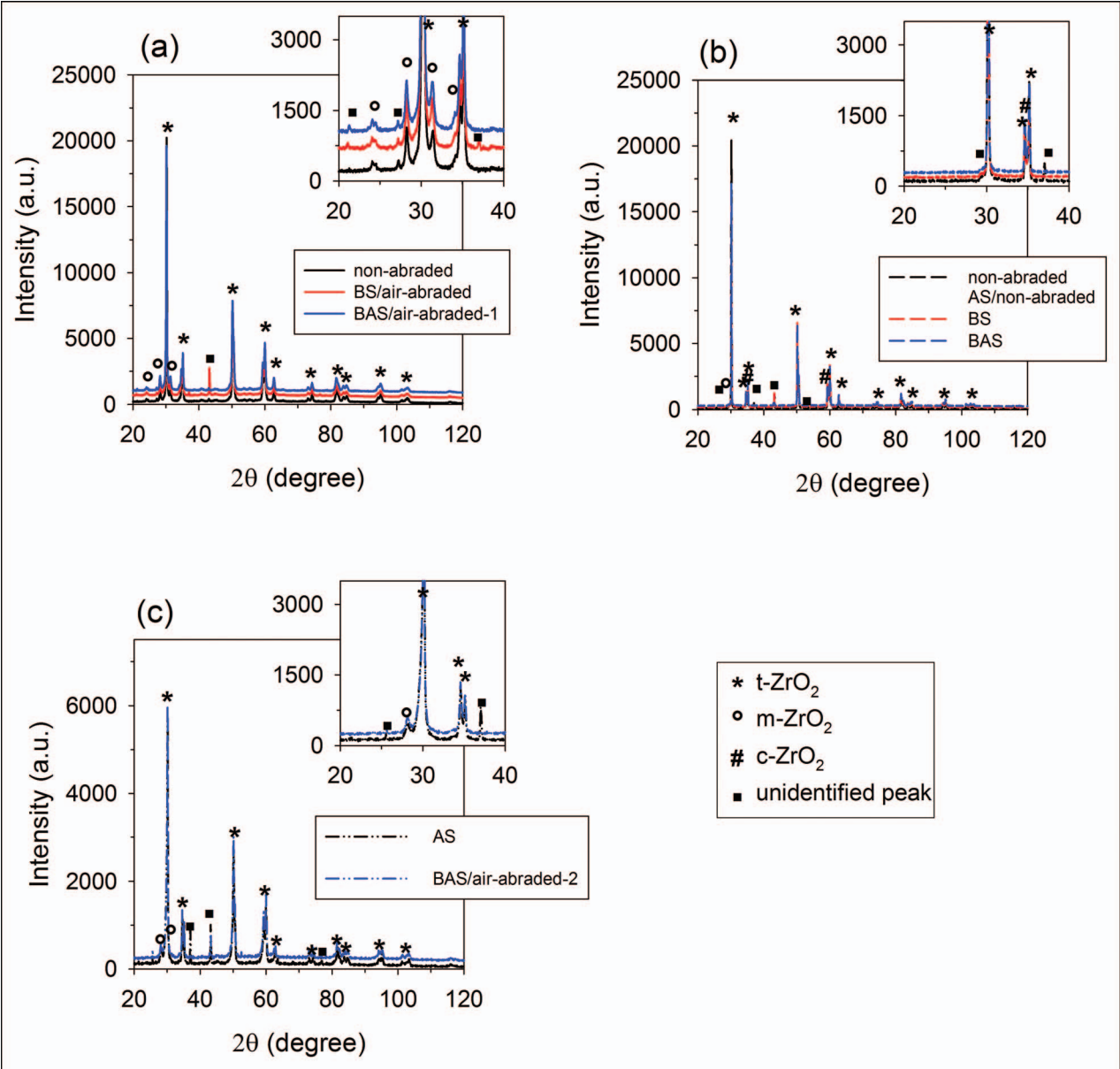


Figure 2. Diffraction patterns of the experimental groups according to each step performed to obtain them. (a): Nonsintered (nonabraded; BS/air abraded; BAS/air abraded 1). (b): Sintered (nonabraded; AS/nonabraded; BS; BAS). (c): Abraded after sintering (AS; BAS/air abraded 2).

for each surface treatment and resin cement. Similarly, in the study of Oyagüe and others,<sup>28</sup> although a correlation analysis was not performed, by observing the results of the zirconia roughness and microtensile bond strength, it seems that there is no correlation between these two variables.

Concerning flexural strength, the AS (1249.1 MPa) and BAS (1181.4 MPa) groups presented higher FS (with no significant difference between them) than the nonabraded (926.8 MPa) and BS (816.3 MPa) groups (with no significant difference between them). Although the BS group exhibited the lowest SBS value, it did not exhibit a decrease in the FS in comparison with the nonabraded group. It is possible that if a bonding agent (silane or adhesive monomers) had been applied after sintering, the wettability of the zirconia by the cement would be improved, resulting in higher SBS values.<sup>11,15,37</sup>

The XRD analysis performed after abrasion for the BS group revealed a monoclinic phase content of 16.3 wt%; however, after sintering, this percentage was zero. For this same condition, Moon and others<sup>24</sup> reported a monoclinic phase content of 16.9 wt% after abrasion, which dramatically decreased to almost zero after sintering. According to these authors,<sup>24</sup> this behavior may be explained by the fact that air abrasion itself induced tetragonal to monoclinic transformation, but a reverse transformation (monoclinic to tetragonal) occurred during the sintering process. Regarding the statistical FS superiority of the AS and BAS groups, it was probably due to the air-abrasion step. These two groups presented higher percentage values of monoclinic phase (AS=12.2 wt% and BAS=8.6 wt%) in comparison with the nonabraded (0.0 wt%) and BS (0.0 wt%) groups. Using 50- $\mu\text{m}$   $\text{Al}_2\text{O}_3$  particles, Monaco and others<sup>22</sup> and Moon and others<sup>24</sup> observed 10.0 wt% and 11.4 wt% of monoclinic phase for the group abraded after sintering. It is known that air abrasion creates surface microcracks around which the grains exhibit a volumetric increase resulting from the tetragonal to monoclinic phase transformation. This outward expansion due to a plastic deformation of the surrounding zirconia provides compressive stresses that counteract the crack propagation.<sup>38</sup> This process, known as transformation toughening, may increase the bulk strength of zirconia,<sup>6,21,27,39,40</sup> as indicated by this study. Although this study did not evaluate the existence of a possible correlation between phase transformation (tetragonal to monoclinic) and flexural strength, it seems that there is some relation between them. Some studies<sup>21,27,40</sup> concluded that

the increase in the mechanical performance of the zirconia seems to be related to the phase transformation (toughening mechanism), given that a higher amount of monoclinic  $\text{ZrO}_2$  content resulted in higher flexural strength values.

Besides the lack of studies that compared the air abrasion routinely performed (AS group) with that performed before sintering (BS group), the novelty of the current research is that the combination of both was tested and yielded the best results with regard to the short-term shear bond strength and flexural strength. However, a concern that should be taken into account is the behavior of the undesirable microcracks in the three air-abrasion conditions. In the BS group, microcracks are created by air abrasion, resulting in a phase transformation (tetragonal to monoclinic) that contains their propagation. After sintering, an inverse phase transformation occurred (monoclinic to tetragonal),<sup>24</sup> releasing the compressive stresses,<sup>22,40</sup> which is not so damaging given that the zirconia has a sintering shrinkage of about 20%-25%, which could promote a partial or total sealing of the cracks.<sup>41</sup> This fact may explain the similar behavior concerning the FS of the BS group in comparison with the nonabraded one. Although the BS group exhibited lower FS than the AS and BAS groups, in the long term it may behave more favorably under cyclic load and moisture. On the other hand, in the AS (the air abrasion routinely performed) and BAS groups, the microcracks created by the air abrasion after sintering were probably contained by the compressive stresses resulting from the phase transformation (tetragonal to monoclinic).<sup>21,27,39,40</sup> This fact may explain the higher FS of these groups in comparison with the BS one. However, we wonder whether the condition of the AS and BAS groups is maintained for a sufficiently long period of time under the adverse effects of the oral environment.

Regardless of choosing the BAS method, which revealed the highest SBS and FS values, or the BS one, which could be more interesting if we consider its supposed long-term mechanical performance, the air-abrasion step performed before zirconia sintering is clinically viable regarding the micromechanical retention. However, in this study, instead of zirconia frameworks, geometrical specimens were used and no attention was given to the potential damage that the air abrasion performed previous to zirconia sintering may cause mainly to the margins of the restorations. Another concern is when this procedure is performed with silica-coated  $\text{Al}_2\text{O}_3$  particles, the chemical bond, which was not the focus of this study,



could be impaired by the surface contamination during the clinical/laboratory steps. Therefore, further research must be carried out investigating other aspects related to the subject of the current study and how these conditions resulting from the air-abrasion/zirconia sintering order behave in a long-term moisture environment, which favors the propagation of microcracks due to the low temperature degradation phenomenon, and under cyclic loading in order to simulate the adverse conditions of the oral cavity.

### CONCLUSIONS

Within the limitations of this *in vitro* study, the following conclusions may be drawn:

1. The rougher surface provided by the air abrasion before zirconia sintering may have impaired the bonding with the resin cement.
2. The morphological patterns resulting from the air-abrasion/zirconia sintering order were consistent with the surface roughness.
3. Considering the short-term adhesive bonding and flexural strength, the air abrasion before and after zirconia sintering, when used in combination, exhibited the best performance.
4. Air abrasion, regardless of the order in which it is performed, provides tetragonal to monoclinic transformation, while sintering tends to zero the monoclinic phase content.

### Conflict of Interest

The authors 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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