



# *In vitro* shear bond strength between fluorinated zirconia ceramic and resin cements

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**PURPOSE.** The purpose of this study was to evaluate the efficiency of a gas-phase fluorination method under different fluorination periods through using two resin cements. **MATERIALS AND METHODS.** 84 zirconia specimens in dimensions of 5 mm × 5 mm × 2 mm were prepared and surface treated with 50 µm aluminum oxide particles or gas phase fluorination for 2 min, 5 min, or 10 min. One specimen in each group was surface analyzed under scanning electron microscope. The remaining specimens were bonded to composite cylinders in dimensions of 2 mm diameter and 3 mm high with Panavia SA Plus or Variolink N. Then, the specimens were stored in 37°C distilled water for 24 hours and shear bond strength test was applied at a speed of 1 mm/min.

**RESULTS.** The highest shear bond strength values were observed in the samples fluorinated for 5 minutes and cemented with Panavia SA Plus. Variolink N did not elicit any statistical differences between surface treatments. Panavia SA Plus resin cement and Variolink N resin cements featured statistically significant difference in shear bond strength values only in the case of 5 minutes of fluorination treatment. **CONCLUSION.** According to the results of this study, application of 5 minutes of fluorination with 10-methacryloyloxydecyl dihydrogen phosphate monomer (MDP) containing Panavia SA Plus resin cement increased the resin bond strength of zirconia. Fluorination of the zirconia surface using conventional resin cement, Variolink N, did not lead to an increase in bond strength. [*J Adv Prosthodont 2018;10:205-10*]

**KEYWORDS:** Zirconia; Resin cement; Airborne-particle abrasion; Gas phase fluorination; 10-methacryloyloxydecyl dihydrogen phosphate monomer (MDP); Scanning electron microscope (SEM)

## INTRODUCTION

Although zirconia restorations are increasingly preferred for dental rehabilitation, they have the limitation of poor resin

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bonding because of their inertness and low surface energy.<sup>1</sup> The glass phase does not exist in zirconia structure, which makes zirconia resistant to hydrofluoric acid.<sup>2-4</sup> For this reason, conventional surface preparation methods are ineffective for zirconia ceramics<sup>3,4,5-8</sup> and alternative techniques are necessary.<sup>5</sup> Various surface treatment methods, such as airborne-particle abrasion,<sup>9-11</sup> selective infiltration etching,<sup>9,10,12</sup> silica-coating methods,<sup>9-11</sup> application of lasers,<sup>9-11</sup> and nano-alumina coating<sup>10</sup> have been developed to yield strong resin bonding to the zirconia surface. The resin bond strength of zirconia changes by several factors, such as surface treatment and wettability of resin cement.<sup>13</sup> Micromechanical lock and/or chemical bonding is essential for strong resin bonding.<sup>6,7,14,15</sup> To achieve micromechanical bonding, the zirconia surface must be roughened.<sup>9</sup> Aggressive mechanical surface abrasion methods that can lead to surface flaws and reduce material strength are required to roughen the zirco-

nia surface.<sup>14</sup> Airborne-particle abrasion with aluminum oxide particles ( $\text{Al}_2\text{O}_3$ ) and application of phosphate ester monomers containing primers are generally employed for resin bonding of zirconia ceramics.<sup>1</sup> Airborne-particle abrasion with  $\text{Al}_2\text{O}_3$  particles decreases surface tension and enhances surface wettability.<sup>16</sup>

The 10-methacryloyloxydecyl dihydrogen phosphate monomer (MDP) is known to increase the bond strength of zirconia ceramics, especially when used with airborne-particle abrasion with  $\text{Al}_2\text{O}_3$  particles or tribochemical coating.<sup>14</sup> Acidic monomers hydrolyze silane-coupling agents and provide siloxane bonds, which are required for chemical bonding. MDP is an acidic monomer and supplies polysiloxane bonding with silane agents, resulting in increased bond strength between zirconia and resin cement.<sup>14</sup>

Plasma treatments are widely used for surface activation of polymers, but they have not been used for zirconia surface activation.<sup>1</sup> Chemically active contents in plasma can increase surface energy without affecting material properties.<sup>1</sup> Gas plasma can create chemically active areas between resin and zirconia.<sup>7,17</sup> Piasek *et al.*<sup>18</sup> developed a gas phase fluorination method based on creating an oxyfluoride conversion layer on the zirconia surface for the purpose of converting the inert zirconia surface to a chemically active surface. With this method, a simple vapor technique is employed to create the oxyfluoride layer.<sup>18</sup> It has the advantage of producing a reactive surface without leading to any physical trauma on the zirconia surface.<sup>19</sup> Previous studies have reported this method to have a positive effect on resin bond strength of zirconia ceramics.<sup>19-21</sup>

Although several methods are reported in the literature in terms of obtaining strong bonding to zirconia restorations, there is still no consensus on the optimal procedure, including bonding agent and surface treatment selection. The purpose of this study was to evaluate the efficiency of fluorination methods and fluorination duration times on

shear bond strength between zirconia and two resin cements by utilizing conventional bisphenol A diglycidylether methacrylate (Bis-GMA)-based and self-adhesive MDP containing resin cements.

## MATERIALS AND METHODS

Experimental materials used in this study and their characteristics are listed in Table 1.

84 zirconia specimens (ICE Zirkon, Zirkozahn, Bruneck, Italy) of dimensions 5 mm × 5 mm × 2 mm were prepared according to the manufacturer's instructions. All specimens were embedded in rectangular acrylic resin blocks that were compatible with the retainer of the universal testing machine for shear bond strength assessment. To provide a standard surface for all specimens, the exposed zirconia surfaces were polished with 400-, 600-, 800-, 1000-, and 1200-grit silicon carbide abrasives (3M ESPE, St. Paul, MN, USA). The specimens were randomly divided into four groups and surface treatments were performed. The plasma treatment was carried out with a RF/LF plasma system (Diener electronic GmbH & Co. KG, Nagold, Germany) using a RF reactor operating at 13.56 MHz. Samples were placed in an plasma reactor chamber and the vacuum chamber was initially evacuated up to  $2 \times 10^{-5}$  MPa and filled with  $\text{SF}_6$  gas. The plasma discharge power was set to 100 watt. The samples were exposed to plasma treatment for different time periods.

Group I: Specimens were airborne-particle abraded with 50  $\mu\text{m}$   $\text{Al}_2\text{O}_3$  particles (BEGO Korox, Bremen, Germany) perpendicular to the specimen surface at 0.28 MPa pressure, 10 mm distance for 20 seconds.

Group II: Specimens were fluorinated for 2 minutes.

Group III: Specimens were fluorinated for 5 minutes.

Group IV: Specimens were fluorinated for 10 minutes.

**Table 1.** Experimental materials and their characteristics

Product	Composition	Manufacturer
Zirconia	$\text{ZrO}_2$ ; specifications, $\text{Y}_2\text{O}_3$ % 4-6, $\text{Al}_2\text{O}_3$ % 1, $\text{SiO}_2$ % max. 0.02, $\text{Fe}_2\text{O}_3$ % max. 0.01, $\text{Na}_2\text{O}$ % max. 0.04	Zirkozahn, Bruneck, Italy
Panavia SA Cement Plus	Paste A: MDP/Bis-GMA/ TEGDMA/HEMA, Hydrophobic aromatic dimethacrylate, Silanated barium glass filler, silanated colloidal silica, dl-Camphorquinone, peroxide, catalysts, pigments Paste B: Hydrophobic aromatic dimethacrylate, Hydrophobic aliphatic dimethacrylate, silanated barium glass filler, surface treated sodium fluoride, accelerators, pigments	Kuraray Noritake Dental Inc., Okayama, Japan
Variolink N	Bis-GMA, urethane dimethacrylate, triethylene glycol dimethacrylate, inorganic fillers: barium glass, ytterbium trifluoride, Ba-Al fluorosilicate glass, spheroid mixed oxide, additional contents: initiators, stabilizers and pigments.	Ivoclar Vivadent AG, Schaan Liechtenstein
Tetric N-Ceram	Urethane dimethacrylate, ytterbium trifluoride, ethoxylated bisphenol A dimethacrylate, Bis-GMA	Ivoclar Vivadent AG, Schaan, Liechtenstein

MDP;10-methacryloyloxydecyl dihydrogen phosphate monomer; Bis-GMA, bisphenol A-glycidyl dimethacrylate; TEGDMA, triethylene glycol dimethacrylate; HEMA, 2-hydroxyethylmethacrylate.

The surface of the one specimen from each group was sputter-coated with gold and analyzed with scanning electron microscopy (SEM) (QUANTA 400F Field Emission SEM) under 10,000 magnification. The remaining specimens were cemented to composite resin cylinders (Tetric N-Ceram: Ivoclar Vivadent AG, Schaan, Liechtenstein) with a diameter of 2 mm and height of 3 mm with Panavia SA Plus (Kuraray Noritake Dental, Okayama, Japan) or Variolink N (Ivoclar Vivadent AG, Schaan, Liechtenstein) according to the manufacturer's protocol. Cemented specimens were stored in 37°C distilled water for 24 hours. Next, shear bond strength testing was performed with the universal test machine (Lloyd-LRX, Lloyd Instruments, Fareham, UK) at a crosshead speed of 1 mm/min.

All data were analyzed using the Statistical Package for the Social Sciences, version 16.0 (SPSS Inc., Chicago, IL, USA). The normality of the data was evaluated with the Shapiro-Wilk test ( $P > .01$ ). Data were statistically analyzed by two-way ANOVA for all groups ( $P < .05$ ) with shear bond strength as the dependent variable and the surface treatments and resin types as independent factors. Multiple comparisons were made with Tukey HSD test.

**Table 2.** Shear bond strength values in MPa

Groups	Resin cement	Mean ± SD (MPa)	n
I	Panavia SA Plus	10.1 ± 5.1 <sup>ab</sup>	10
	Variolink N	6.1 ± 2.0 <sup>a</sup>	10
II	Panavia SA Plus	7.6 ± 3.2 <sup>ab</sup>	10
	Variolink N	7.9 ± 2.4 <sup>ab</sup>	10
III	Panavia SA Plus	16.3 ± 3.1 <sup>c</sup>	10
	Variolink N	6.8 ± 2.6 <sup>a</sup>	10
IV	Panavia SA Plus	12.7 ± 5.8 <sup>bc</sup>	10
	Variolink N	7.6 ± 3.9 <sup>ab</sup>	10

\*The same superscript letters have no statistically significant difference.

## RESULTS

The shear-bond strength values are listed in Table 2.

According to the two way ANOVA results, there are statistical differences between the surface treatments and resin cements ( $P < .05$ ).

Specimens cemented with Panavia SA Plus did not exhibit statistically significant differences among Group I, II, and IV, while Group III had the highest shear-bond strength values. There was no statistical difference between Group III and IV.

Specimens cemented with Variolink N had statistically similar results among all groups.

In Group I, II, and IV, there were no statistically significant differences between the two resin cement used.

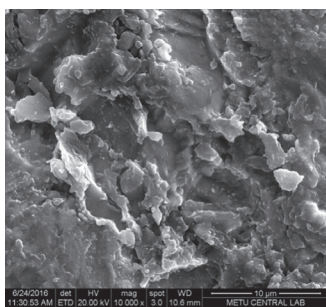
Group III with Panavia SA Plus cement had statistically higher shear bond strength values than with Variolink N.

SEM images of each group are shown in Fig. 1, Fig. 2, Fig. 3, Fig. 4.

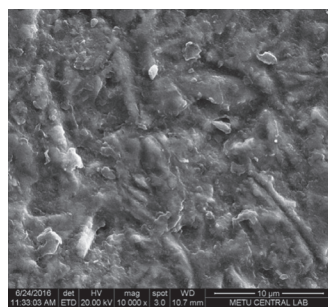
Deep irregularities and sharp edges were observed in the SEM evaluation of the airborne-particle abraded zirconia surface. Fluorination treatment resulted in a smoother zirconia surface compared to airborne-particle abrasion. More regular surface roughness was observed after 2 and 5 minutes of fluorination. 10 minutes fluorination led to a generally stretched surface topography and somewhat deep grooves.

## DISCUSSION

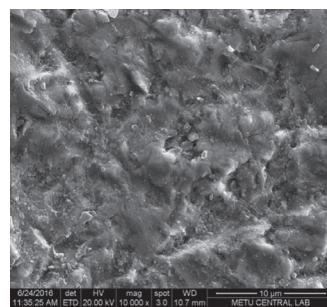
This study compared the generally used zirconia surface treatment method of airborne-particle abrasion with  $Al_2O_3$  particles and the gas-phase fluorination method for resin bonding of zirconia ceramics with two resin cements - MDP containing Panavia SA Plus and a conventional resin cement Variolink N. The results indicated that resin cement selection is an important factor when fluorination is used as a surface treatment method. The fluorinization time applied also affected bond strength. The highest shear-bond strength was obtained with 5 minutes of fluorination combined with the use of Panavia SA Plus.



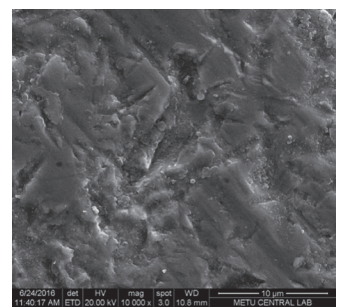
**Fig. 1.** Airborne-particle abrasion with 50 μm  $Al_2O_3$ .



**Fig. 2.** 2 min gas-phase fluorinated zirconia surface.



**Fig. 3.** 5 min gas-phase fluorinated zirconia surface.



**Fig. 4.** 10 min gas-phase fluorinated zirconia surface.

It is questionable to use *in vitro* test methods to develop an idea of the clinical performance of dental adhesives because it is difficult to generalize laboratory data to clinical conditions.<sup>22</sup> The macro bond strength tests (shear and tensile tests) have the problem of large bonding area, varying from 7 mm<sup>2</sup> to 28 mm<sup>2</sup>, which leads to inhomogeneous stress distribution, higher coefficient variation, and lower percentage of adhesive failures.<sup>22</sup> For this reason, a relatively smaller surface area was employed in this study for shear testing to obtain a more homogenous bonding interface, including reduced flaws compared to larger areas. Although shear test has disadvantages, it features the benefits of easy application procedure, minimal equipment requirements, and a ready specimen preparation procedure, which makes it popular.<sup>22</sup>

Airborne-particle abrasion with Al<sub>2</sub>O<sub>3</sub> has been reported as an effective method for obtaining stable resin bonding to zirconia ceramics.<sup>23</sup> It offers a roughened surface with elevated surface energy and wettability.<sup>24</sup> Increased bond strength with airborne-particle abrasion has been described in the literature.<sup>11,24-26</sup> Airborne-particle abrasion was selected as the control method for this work because it provides increased bond strength and is routinely used with zirconia ceramics. Ito *et al.*<sup>26</sup> showed that XRD analyses of airborne-particle abraded zirconia indicated that airborne-particle abrasion forms microcracks on the zirconia surface and causes a phase transition, and this is supported by other studies.<sup>27-29</sup> Phase transition leads to formation of residual compressive strengths and improvement in mechanical properties.<sup>27,28</sup> However, larger blasting particles induce deeper monoclinic phase transformation, reducing flexural strength.<sup>27</sup> For this reason, 50 μm Al<sub>2</sub>O<sub>3</sub> particles were utilized herein to prevent deeper phase transformation.

With a gas-phase fluorination method, fluorinated plasma (SF<sub>6</sub> gas) is applied to the zirconia surface, converting it into zirconium oxyfluoride (ZrO<sub>x</sub>F<sub>y</sub>).<sup>18,20</sup> It was previously demonstrated that fluorinization treatment was effective for either polished or roughened surfaces,<sup>18</sup> and this can be considered an advantage to eliminating the airborne-particle abrasion procedure. Gas-phase treatment was applied for 2 minutes, 5 minutes, and 10 minutes in this study. The application period did not influence the bond strength of the groups cemented with Variolink N while it notably affected the bond strength of the groups cemented with Panavia SA Plus. 2 minutes fluorination did not enhance bond strength. 5 minutes fluorination with Panavia SA cement resulted in the highest bond strength. However, bond strength did not rise proportionally with application time. Although 10 minutes fluorination with Panavia SA Plus had statistically similar shear-bond strength values to 5 minutes fluorination with use of Panavia SA Plus, the obtained bond strength values were lower. This may have been because of thicker layer of oxyfluoride at the zirconia-resin interface, which makes the bond less stable and resistant.

It was reported<sup>18,20,21,30</sup> that fluorination of zirconia improved surface wettability and bonding ability of zirconia ceramics. Piascik *et al.*<sup>18,20</sup> observed that 2 minutes fluorina-

tion led to higher bond strength values than airborne-particle abrasion. The results of the current study are not in agreement. 2 minutes fluorination did not result in any enhancement in bond strength with either resin cements used here. This might have been based on different composition, solvent, initiator, viscosity and wettability of the bonding agents used in Piascik *et al.*'s studies. In addition, varied parameters used in the plasma treatment could have been a factor.

The oxyfluoride surface obtained via gas-phase fluorination reacts with organo-silanes<sup>18</sup> and ester groups in acrylate-based monomers.<sup>21,30</sup> It was demonstrated that fluorination improved the bond strength with or without organosilanes.<sup>21</sup> The results of this study are partially in agreement. Only 5 minutes of fluorination combined with use of Panavia SA Plus improved bond strength.

Adhesive strategies that combine chemical and mechanical surface treatment methods have been developed.<sup>31,32</sup> MDP is a popular and widely used phosphate monomer that chemically bonds to hydroxyl groups of metal ions that exist on the ceramic surface.<sup>32</sup> It achieves bifunctional adhesion with Bis-GMA and metal oxides.<sup>33,34</sup> It is known that MDP monomers containing bonding agents enhance the bond strength of airborne-particle abraded zirconia restorations.<sup>31,32,35-37</sup> In this study sandblasted samples did not show statistical difference between the resin cements used however MDP based resin cement numerically increased the bond strength (Panavia SA Plus; 10.1 ± 5.1, Variolink N; 6.1 ± 2). This can be explained with the higher surface wettability of airborne-particle abraded surfaces and chemical affinity of the MDP monomer.

In this study, Bis-GMA-based resin cements, Panavia SA Plus cement and Variolink N, were used for the purpose of evaluating the efficiency of a gas-phase fluorination method. Although both of the resin cements were Bis-GMA-based, they had different compositions. The major difference is that Panavia SA Plus cement includes MDP monomers with an affinity to zirconium oxide. This makes it possible to understand the behavior of MDP monomer in combination with a fluorinated plasma treatment. The obtained results indicate that using conventional bis-GMA-based resin cement is ineffective with fluorination treatment. Yet, MDP and silanated monomers containing Panavia SA Plus can be employed with 5 minutes of fluorination, and this may be explained through the reaction between phosphate esters and the fluorinated zirconia surface. The fluorinated surface becomes hydrolyzed when exposed to atmosphere, where hydroxyl groups of the phosphate ester-containing monomers in the adhesive react with the hydrolyzed surface.<sup>21</sup>

The surface of one specimen from each group was evaluated under SEM. Evaluation of the airborne-particle abraded zirconia surface indicated that airborne-particle abrasion leads to irregular surface roughness formation, and this is in agreement with previous studies.<sup>4,11,24,31,36</sup> Fluorinated surfaces were smoother than airborne-particle abraded surfaces. Fluorination time affected the surface morphology; the 2

minutes- and 5 minutes-fluorinated zirconia surfaces had quite similar roughness, while the 10 minutes-fluorinated surface was the smoothest and included deep fissures. The lower bond strength values after 10 minutes of fluorination compared to 5 minutes could be explained by the fact that. Although the 2 minutes-fluorinated zirconia surface was roughened, the lowest bond strength values were obtained in this group. This can be interpreted as 2 minutes of plasma treatment not providing sufficient density for the oxyfluoride layer.

In the present study, the shear-bond strength test was applied following 24 hours of water storage in distilled water so the obtained results cannot be generalized to long-term clinical performance, so further studies are needed for long-term evaluation. Additionally, combining the fluorinization method with other bonding agents will be helpful for having a more generalized understanding of the bonding behavior to fluorinated surfaces, which is still unclear.

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