Effect of surface treatments on the bonding strength of self-adhesive resin cements to zirconia ceramics

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Objective: To investigate the effect of surface treatments on the surface properties of yttria-stabilized zirconia ceramics (Y-TZP) and the interfacial bond strength to self-adhesive resin cements. Method and Materials: Two types of Y-TZP (Vita In-Ceram YZ [VZ] and IPS e.max ZirCAD [IZ]) were used. The specimens were divided into four groups in each test according to the surface treatment used: Group A (control; no treatment), Group B (airborne-particle abrasion), Group C (CH₃Cl₂ for 60 minutes), and Group D (hot etching for 60 minutes). Scanning electron microscopy, atomic force microscopy, and X-ray diffractometry were carried out. Two types of self-adhesive resin cements (i CEM [IC] and VM9) were measured with a four-point bending configuration. Following fracture testing, specimens were examined with a stereomicroscope. Data were analyzed using ANOVA and Tukey test. Results: IZ treated with hot etching showed the highest average surface roughness values (172.5 ± 15.43 nm) compared with the other groups (P < .05). The greatest amount of monoclinic phase was measured after airborne-particle abrasion for VZ (8.9%), followed by IZ (6.1%). Improvements in bond strength values were found in the following order: hot etching > CH₃Cl₂ > airborne-particle abrasion > no treatment. Most failure modes were adhesive type of failures between ceramics and cement material (68.76%). Conclusion: Adhesion between Y-TZP and self-adhesive resin cements can be improved by the use of CH₃Cl₂ or hot etching surface treatments prior to resin cement application as an alternative technique to airborne-particle abrasion treatment. (Quintessence Int 2013;44:e170–e180; doi: 10.3290/j.qi.a29572)

Key words: adhesion, atomic force microscopy, self-adhesive resin cement, surface treatments, X-ray diffractometry, Y-TZP ceramic

In recent years, there has been widespread use of zirconia (ZrO₂) ceramics in dentistry, with a high popularity in the field of prosthodontics.¹-³ ZrO₂ provides superior strength and fracture toughness compared to traditional porcelain and other silica-based materials, while presenting improved esthetic properties compared to metallic-based prosthetics.⁴ However, the highly crystalline structure of ZrO₂ ceramic creates difficulties for bonding ZrO₂ with dental resin cements.²,⁵ The absence of silica or any considerable glassy phase in the microstructure of ZrO₂ eliminates the capability of conventional etching as a method to roughen the surface for improving mechanical bonding, and impedes the use of traditional silane as there is no silica present to readily form surface hydroxyls for chemical bonding.⁴,⁶,⁷ To overcome this problem with ZrO₂–resin cement bonding, a number of surface modification methods have been proposed to improve adhesion properties of ZrO₂ in an effort to enhance bonding between ZrO₂ and resin cements. They included selective infiltration etching technique, plasma spraying, coating with zirconia ceramic powder, laser treatment, silica coating, airborne-particle abrasion using alumina or other abrasive particles, and surface fluorination, with varying results regarding bond strength.²,⁸-¹⁴ As yet, there is no agreement concerning the efficient surface treatment method for achieving optimal bond strength between ZrO₂ ceramics and resin cements. It is important to improve the bond quality

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and strength between resin cements and ZrO₂ ceramics, as these are the key factors in the success of resin-bonded fixed partial dentures (FPDs).² Surface roughening of ZrO₂ surface by airborne-particle abrasion does not always improve the adhesion between ZrO₂ and resin cements.⁶,¹⁵ In addition, airborne-particle abrasion can trigger the transformation from tetragonal to monoclinic structure, which can decrease the fracture strength of ZrO₂ due to the missing transformation capability during critical loading, and may also damage the long-term lifespan of ZrO₂ ceramics.¹⁶,¹⁷ Consequently, a nondestructive, simple, and applicable method for pretreating ZrO₂ ceramic surfaces would be clinically beneficial.¹⁸

The nature of bonding between ZrO₂ ceramics and resin cements requires further investigation. The development of adequate surface treatment of ZrO₂ ceramics will enhance the adhesion and the success rate of FPDs. Recently, an experimental hot chemical etching solution, previously used for conditioning metal and/or alloys¹⁹ has been applied on ZrO₂ ceramics, with the result of improving average surface roughness and enhancing the bond strength with the resin cement.²⁰,²¹ It would be beneficial to find chemical baths that could enhance the bond strength between ZrO₂ ceramics and resin cements. Methylene chloride has been applied for conditioning the surface of titanium for porcelain bonding with improvement in adhesion.²² The organic solvent, methylene chloride solution, has not been evaluated for its effect on the adhesion of resin cements to ZrO₂ ceramics. It is not known how surface treatment of ZrO₂ ceramics using certain chemical baths (such as methylene chloride and experimental hot chemical etching solution) could lead to better bond strength or adhesion results.

Consequently, the aim of this study was to determine the effect of different surface treatments on the surface properties of yttria-stabilized zirconia ceramics (Y-TZP) and to investigate the outcomes of these treatments on the adhesion of different Y-TZP to resin cements. The null hypothesis is that the proposed surface treatments do not affect the surface properties of Y-TZP substrate and the adhesion between Y-TZP and resin cements.

**METHOD AND MATERIALS**

**Specimen preparation**

Two hundred and eighty specimens of two types of Y-TZP ceramic blocks (Vita In-Ceram YZ [VZ], Vita Zahnfabrik; and IPS e.max ZirCAD [IZ], Ivoclar-Vivadent) were used for this study. The Y-TZP specimens with the required dimensions for each test were cut from pre-sintered blocks using a water-cooled low-speed diamond saw (Isomet, Buehler). The Y-TZP specimens were cut 20% larger than the desired dimension considering shrinkage, and sintered at 1,530°C for 2 hours in the Vita Zyrcomat furnace (Vita Zahnfabrik). Then, the specimens were wet ground using 600-grit silicon carbide (SiC) paper (Leco) and then cleaned in an ultrasonic bath (Sonorex, Branson) for 20 minutes.

The specimens in each test were divided into four groups according to the surface treatment that was performed, as follows:

- **Group A – Control (no treatment):** the specimens were exposed only to grinding and ultrasonic cleaning as mentioned above
- **Group B – Airborne-particle abrasion:** the specimens were abraded with Al₂O₃ particles (110 µm) with a dental airborne-particle abrasion instrument (Micro-Blaster, Daedong Industrial) that was applied for 20 seconds at a pressure of 2 bar with a distance of 15 mm between the nozzle and the surface.
- **Group C:** the specimens were immersed in methylene chloride (CH₂Cl₂, formula weight 84.13 g/mol; El Nasr Pharmaceutical Chemicals) for 60 minutes.
- **Group D – experimental hot etching solution:** a solution with 800 ml of methanol, 200 ml of 37% HCl, and 2 g of ferric chloride (FeCl₃) was heated up to 100°C in a water bath during the etching process.¹⁹ The Y-TZP specimens were immersed in the solution for 60 minutes.²⁰

Treated specimens were rinsed with distilled water and then ultrasonically cleaned in a deionized water bath for 10 minutes and gently air dried.

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Scanning electron microscopy (SEM)
Twelve square-shaped specimens (10 mm × 10 mm × 1 mm) from each type of the sintered treated Y-TZP plates (n = 3/group) were rinsed with 96% ethanol and air dried, mounted on metallic stubs, sputtered (Sputter Coater S150A) with a gold layer and then examined under SEM (JEOL JXA-840A, JEOL) at 15,000× magnification, to observe the features of the treated surfaces.

Atomic force microscopy (AFM)
Twenty-eight square-shaped specimens (10 mm × 10 mm × 1 mm) from each type of Y-TZP (n = 7/group) were used to characterize the surface microtopography of the sintered treated Y-TZP plates by contact-mode AFM (Autoprobe CP-II, Veeco). The radius of curvature of the scanning tip was 10 nm. Images were recorded with a scan rate of 1 Hz at a resolution of 512 × 512 pixels per image and scanning area of 10 μm × 10 μm. The average surface roughness \( R_s \) of the Y-TZP specimen after different treatments was recorded in nanometers (nm). For each specimen, the surface roughness was measured at three different sites and the \( R_s \) was then calculated. \( R_s \) values were compared with two-way ANOVA considering two factors (surface treatment and type of Y-TZP) and their interactions. Multiple comparisons were made by Tukey test. Statistical significance was set at the .05 probability level.

X-ray diffractometry (XRD)
Four square-shaped specimens (10 mm × 10 mm × 1 mm) from each type of the sintered treated Y-TZP plates were analyzed by X-ray diffractometry (X’Pert Pro, Panalytical) to determine the crystal phases of the sintered Y-TZP grains to quantify the monoclinic and tetragonal phases. Specimen surfaces were scanned with Cu Kα X-ray over an angular range of 20 to 65 degrees in 2θ degrees with a step size of 0.05 degrees and 5-second step interval. The relative amount of the monoclinic phase \( X_M \) was calculated as suggested by Garvie and Nicholson:23

\[
X_M = \frac{I_{M(111)} + I_{M(110)}}{I_{M(111)} + 2I_{M(110)} + I_{T(101)} + I_{T(111)} + I_{M(111)} + I_{M(110)}}
\]

where \( I \) is the intensity of the peak, \( M(111) \) corresponds to the monoclinic phase referring to the crystallographic plane (111), \( T(101) \) corresponds to the tetragonal phase referring to the crystallographic plane (101) and \( M(111) \) corresponds to the monoclinic phase referring to the crystallographic plane (111).

Y-TZP/resin adhesion test
Ninety-six rectangular sintered specimens from each Y-TZP material were divided into four groups according to the method of surface preparation (n = 24/group). Each group was further equally divided into two subgroups according to the type of self-adhesive resin cement used in this study (n = 12/subgroup). Two types of self-adhesive resin cements were used: iCEM (IC) dual-curing (Heraeus Kulzer) and Multilink Speed (MS) self-curing (Ivoclar Vivadent). A split Teflon mold (30 mm × 8 mm × 1.5 mm) was used to apply resin cements onto the Y-TZP specimen.22,24 The resin cements were prepared according to their manufacturer’s instructions. For IC dual-curing resin cement, the specimens were light polymerized in intervals of 40 seconds using an overlapping procedure for a total of 200 seconds with a halogen light-curing unit (XL2500, 3M ESPE) with an output of 670 mW/cm². After setting of the cement, the Teflon mold was disassembled (Fig 1).

All specimens were subjected to thermocycling between 5°C and 55°C for 10,000 cycles with a 30-second dwell time. After that, the specimens were notched...
across the width and entirely through the depth of the cement layer at the middle of the specimen using a water-cooled low-speed diamond saw which gave an accurate notch of width 0.4 mm and a depth of 1.5 mm (the thickness of cement). A pre-crack was created at the Y-TZP–resin interface in a three-point bending jig, with a total length of approximately 2 mm.\(^\text{25}\)

The precracked specimens were then tested in a universal testing machine (TT-B, Instron) using a four-point bending jig with the inner rollers 14 mm and the outer rollers 26 mm at a crosshead speed of 0.05 mm/minute until the crack reached the inner rollers. The plateau load at which stable crack extension occurred was collected for calculation of the strain energy release rate, \(G\).

The testing configuration shown in Fig 1 illustrates the relationship between the specimen and the rollers of the four-point bending jig.

### Adhesion determination

The strain energy release rate, \(G\), is given by:\(^{26}\)

\[
G = \frac{\eta \left[ P^2 (1 - \nu_r^2) \right]}{E_r (h_r)^3}
\]

where \(P\) is the load to stably propagate the crack, \(l\) the moment arm or distance between inner and outer load line (rollers) on the same side, \(\nu_r\) and \(E_r\) are Poisson's ratio and elastic modulus of Y-TZP substrate (VZ = 0.32, 210 GPa; and IZ = 0.3, 200 GPa) respectively, and \(b\) and \(h\) are the specimen width and total thickness, respectively.

The nondimensional parameter \(\eta\) is calculated with the all-dimensional parameters by:

\[
\eta = \frac{3}{2} \left[ \frac{1}{(\nu_r)^2} - \frac{1}{\left(\frac{1}{\nu_r^2} + \frac{1}{\nu_r^2 + \frac{1}{2(1-\nu_r^2)}}\right)^2} \right]
\]

with

\[
\lambda = \frac{E_r (1 - \nu_r^2)}{E_c (1 - \nu_c^2)}
\]

where \(\nu_r\) and \(E_r\) are Poisson’s ratio and elastic modulus of resin cement (IC = 0.3, 4.92 GPa; and MS = 0.27, 4 GPa), and \(h_r\) and \(h_z\) are thickness of resin cement and Y-TZP substrate respectively. A statistical analysis of the strain energy release rate (G) values was analyzed using three-way ANOVA considering three factors (surface treatment, type of Y-TZP, and type of resin cement) and their interaction. Multiple comparisons were made by Tukey test. Statistical significance was set at the .05 probability level.

Following the interfacial fracture test, failure modes were analyzed with a stereomicroscope (Olympus SZX-ILLB100-Olympus Optical) with 30× magnification, and classified into three categories: Type 1, adhesive failure at the resin–Y-TZP interface; Type 2, cohesive failure inside the resin material; and Type 3, mixed failure.

### RESULTS

Representative SEM and AFM images of the treated VZ and IZ Y-TZP ceramics are presented in Figs 2 and 3, respectively. The surfaces exhibited grain structures with blunt and round edges for both types of untreated groups of Y-TZP ceramics (Figs 2a and 2e). The surfaces of specimens treated by airborne-particle abrasion showed well-defined, micro-sized elevated and depressed areas with groove-shaped micoretentions, which possibly resulted from the high impact of blasting particles (Figs 2b, 2f, 2g, and 2h). Surface treatment with \(\text{CH}_2\text{Cl}_2\) showed more areas of depression on the surface compared with untreated groups (Figs 2c, 2g, 3c, and 3g). Experimental hot etching treatment of the Y-TZP resulted in a rough and irregular surface with deposition of homogenous coated layer formed on the surfaces of VZ and IZ Y-TZP (Figs 2d, 2h, 3d, and 3h).

Two-way ANOVA of the surface roughness testing data (surface treatment and type of Y-TZP) revealed that the surface roughness was significantly affected by the surface treatment and type of Y-TZP \((P < .001)\). There were significant interactions between surface treatment and type of Y-TZP \((P = .006)\). Means and standard deviations of the treated Y-TZP average surface roughness \((R_a, \text{nm})\) have been extrapolated from the AFM images (Fig 3), and with their significant differences are presented in Table 1. In general, Y-TZP treated with airborne-particle abrasion or experimental hot etching solution for 60 minutes showed significantly the highest \(R_a\).
values compared with the other groups ($P < .05$). However, there was no significant difference between the groups treated with CH$_2$Cl$_2$ for 60 minutes and the control group ($P > .05$) (Table 1).

According to the XRD patterns, the major peak of the tetragonal phase is presented at 29.8 degrees, corresponding to the (101) crystallographic plane as obtained from the XRD standard file 024-1164. The monoclinic phase has the major peaks at 28.03 degrees and 31.36 degrees corresponding to the (111) and (111) crystallographic planes as obtained from the XRD standard file 05-0543 (Fig 4). The greatest amount of monoclinic phase is measured after airborne-particle abrasion for VZ (8.9%), followed by IZ (6.1%). The amount of monoclinic phase is low in the
Table 1: Mean (standard deviation) of the surface average roughness values ($R_a$, nm) of treated Y-TZP ceramics, and statistical differences

<table>
<thead>
<tr>
<th>Y-TZP</th>
<th>Surface treatments</th>
<th>Airborne-particle abrasion</th>
<th>$CH_2Cl_2$</th>
<th>Hot etching</th>
</tr>
</thead>
<tbody>
<tr>
<td>VZ</td>
<td>No treatment</td>
<td>92.78 (8.33)$^{a,b}$</td>
<td>125.43 (10.37)$^{a,b,c,d}$</td>
<td>99.18 (8.31)$^{a,c}$</td>
</tr>
<tr>
<td>IZ</td>
<td>No treatment</td>
<td>124.07 (8.17)$^{b,c,d}$</td>
<td>176.87 (14.73)$^{a,b,c}$</td>
<td>133.78 (10.42)$^{a,b}$</td>
</tr>
</tbody>
</table>

Mean values represented with common or same superscript uppercase letters (row) or lowercase letters (column) are not significantly different according to Tukey test ($P > .05$).
control and the other experimental groups for VZ and IZ Y-TZP ceramics (Fig 5).

Three-way ANOVA of the strain energy release rate (G-value) testing data (surface treatment, type of Y-TZP, and type of resin cement material) revealed that the bond strength was significantly affected by surface treatment, type of Y-TZP, and type of resin cement material ($P < .001$). There were significant interactions between type of Y-TZP and surface treatment, as well as type of resin cement material and surface treatment ($P < .05$). However, there was no significant interaction between type of Y-TZP and resin cement material, as well as type of Y-TZP, surface treatment, and type of resin cement material ($P > .05$). The means of the strain energy release rate ($W_{BMVF + N}$ and standard deviations are presented in Table 2. The results of bond strength values achieved with experimental etching solution for 60 minutes were significantly higher compared with the other groups ($P < .05$). The IZ/IC (etching for 60 minutes) group showed the highest G-value ($12.57 \pm 1.94 \text{ J/m}^2$) among the groups. In general, regarding type of surface treatment, improvements in bond strength values were found in the following order: experimental etching solution > CH$_2$Cl$_2$ > airborne-particle abrasion > no treatment; however, regarding the Y-TZP/resin cement system, improvements in bond strength values were found in the following order: IZ/IC > VZ/IC > IZ/MS > VZ/MS (Table 2). Most failure modes were adhesive types of failure between ceramic and cement material (68.76%). In addition, mixed failures (25%),

**Fig 4** XRD analyses of different experimental groups: (a) VZ and (b) IZ. T, the tetragonal zirconia phase; M, the monoclinic zirconia phase.

**Fig 5** Relative content of monoclinic (%) of different tested groups.
and cohesive failures within the cement (6.24%) were also observed for the differently tested groups (Fig 6).

**DISCUSSION**

In accordance with the results of this study, the null hypothesis was rejected since differences in surface characteristics of Y-TZP substrate and adhesion between Y-TZP and resin cements were found between the tested groups.

Resin bonding to high-strength non-etchable ceramics is not always predictable and needs different bonding methods from silica-based dental ceramics. Consequently, additional in vitro and clinical studies are required. The present study evaluated the effect of various surface treatments on the surface properties and bond strength of Y-TZP to self-adhesive resin cements. In this study, SEM and AFM images showed some differences between treated Y-TZP surfaces, compared to the untreated Y-TZP surface (Figs 2 and 3). It has been reported that roughening of the substrate surface enhances adhesion as it permits the resin cement to flow into the surface, forms irregularities on the substrate surface, and creates micromechanical retention.

Measuring the bond strength of bimaterial interface has been conventionally carried out in shear tests, from which the stress at bond failure was determined. Shear bond strength tests ignore the nature of the stresses created inside the adherence zone, which can have a significant influence on the mode of failure. In addition, most of the bond tests are associated with stress gradients along the interfacial regions and stress concentration effects. An alternative fracture mechanics method in evaluating the adhesion at a bimaterial interface has been described by Charalambides et al. This approach has previously been applied in dentistry to assess interfacial fracture toughness in terms of the strain energy release rate (G-value, J/m²) of various metal-ceramic systems. One of the advantages of this approach is that the adhesion of different systems can be compared directly. In the present study, the adhesion of self-adhesive resin cements to Y-TZP using different surface treatments was determined by the interfacial strain energy release rate (G-value) test. The specimen geometry and the precrack prepared before testing led to the remarkable stability of crack extension along the interface. The measurements can be obtained without the effect of other variables in the luting agent or ceramic substrate.

This study revealed that the application of hot etching solution to Y-TZP surfaces provided the highest G-value bond strength followed by CH₄Cl₂ treatment (Table 2). These results are in agreement with Casucci et al when etching ZrO₂ ceramic using the hot etching solution for improving the micromechanical retention of ZrO₂ substrate. This could be attributed to

**Table 2** Mean (standard deviation) of the strain energy release rate (G-values, J/m²) of resin cements to treated Y-TZP ceramics, and statistical differences

<table>
<thead>
<tr>
<th>Y-TZP/ resin cement materials</th>
<th>Surface treatments</th>
<th>Control</th>
<th>Airborne-particle abrasion</th>
<th>CH₄Cl₂</th>
<th>Hot etching</th>
</tr>
</thead>
<tbody>
<tr>
<td>VZ</td>
<td>IC</td>
<td>4.25 (0.81)</td>
<td>4.61 (0.89)</td>
<td>6.14 (1.25)</td>
<td>9.97 (1.06)</td>
</tr>
<tr>
<td></td>
<td>MS</td>
<td>2.30 (0.67)</td>
<td>2.59 (0.96)</td>
<td>3.88 (0.91)</td>
<td>7.79 (1.40)</td>
</tr>
<tr>
<td>IZ</td>
<td>IC</td>
<td>5.26 (0.59)</td>
<td>5.57 (0.61)</td>
<td>7.02 (1.02)</td>
<td>12.57 (1.94)</td>
</tr>
<tr>
<td></td>
<td>MS</td>
<td>3.02 (0.61)</td>
<td>3.47 (0.49)</td>
<td>5.01 (0.97)</td>
<td>8.19 (2.08)</td>
</tr>
</tbody>
</table>

Mean values represented with common or same superscript uppercase letters (row) or lowercase letters (column) are not significantly different according to Tukey test (P > .05).
the corrosion-controlled process produced by the hot etching solution. The immersion in the hot etching solution may possibly have assisted the breakdown of the protected \( \text{ZrO}_2 \) layer and initiated localized corrosion phenomena. For \( \text{CH}_2\text{Cl}_2 \) treatment, the interfacial bond strength between Y-TZP and self-adhesive resin cements was significantly enhanced compared with the control groups. Although the surface roughness of the Y-TZP treated with \( \text{CH}_2\text{Cl}_2 \) slightly increased compared with the control groups (Table 1), the adhesion between Y-TZP and resin cements was improved (Table 2). This treatment modified the surface of Y-TZP as presented in Fig 2. On the other hand, roughening the Y-TZP surface with airborne-particle abrasion had a slight influence on providing reliable bond strength between the resin cements and Y-TZP (Table 2). This finding is in agreement with previous studies, verifying that it is difficult to achieve a reliable bond between resin cements and inert \( \text{ZrO}_2 \) ceramics without any previous surface treatment.

Selection of the luting cement seems to be a more relevant factor while bonding to \( \text{ZrO}_2 \) ceramics. In the present study, the bond strength value for the IC dual-curing self-adhesive resin cement with the Y-TZP was found to be more efficient than the MS self-curing self-adhesive resin cement (Table 2). It has been reported that the lower bond strength values for the self-acti-
vated luting agents is due to the acidity of the adhesive system used. This is because of the presence of residual acid monomers in the resinous cement layer not polymerized. These residual acid monomers react with tertiary amine, present in the self-activated resinous cement, neutralizing it, so that it is unable to react with the benzoyl peroxide, which is responsible for the setting reaction of the polymerization process of this cement.\(^{43,44}\)

Regarding the type of Y-TZP, the bond strength was significantly affected \((P < .05)\). The higher bond strength was achieved with IZ Y-TZP (Table 2). This could be attributed to the higher surface roughness of IZ Y-TZP compared with VZ ceramic (Table 1). The IZ-surface topography appeared a rather rough surface as revealed by AFM results (Table 1 and Fig 3). It could be speculated that the \(\text{ZrO}_2\) ceramic topography may enhance the micromechanical retention besides the positive effect of \(\text{ZrO}_2\) surface pretreatments.

Failure modes analysis revealed less adhesive failure for \(\text{CH}_3\text{Cl}_2\) or hot etching-treated Y-TZP as compared to the control and airborne-particle abrasion groups (Fig 6). This observation ratifies that sufficient micromechanical retention is not only important for establishing high bond strengths, but also for impeding adhesive failure.\(^{8,45}\)

The present study showed that treatment of Y-TZP surfaces with \(\text{CH}_3\text{Cl}_2\) or hot etching solutions can enhance the bond strength to self-adhesive resin cements, as compared to the untreated surfaces. Further studies are needed to assess the durability of the bond to the \(\text{CH}_3\text{Cl}_2\) and hot etching-treated Y-TZP surface using the materials tested in the present study. Additionally, randomized clinical trials are required to give reliable recommendations for dental practitioners.

**CONCLUSION**

Based on the results presented and within the limitations of this study, the following conclusions were drawn. Methylene chloride or hot etching treatments could be considered as alternative treatment modalities to airborne-particle abrasion for yttria-stabilized zirconia ceramics to enhance adhesion with resin cements and to avoid microcrack formation and phase transition that would be detrimental to the longevity of ceramic restorations.

**REFERENCES**


