Bonding to Zirconia Using a New Surface Treatment

Moustafa N. Aboushelib, DDS, MSc, PhD,[1] Albert J. Feilzer, DDS, PhD,[2] & Cornelis J. Kleverlaan, PhD[2]

[1] Dental Biomaterials Department, Faculty of Dentistry, Alexandria University, Egypt
[2] Dental Materials Science Department, Academic Center for Dentistry Amsterdam (ACTA), University of Amsterdam and the Free University, The Netherlands

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Correspondence
Moustafa N. Aboushelib, Alexandria University, Faculty of Dentistry
Material Science, Azarita Alexandria, Egypt.
E-mail: Bluemarline_1@yahoo.com

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Abstract

Purpose: Selective infiltration etching (SIE) is a newly developed surface treatment used to modify the surface of zirconia-based materials, rendering them ready for bonding to resin cements. The aim of this study was to evaluate the zirconia/resin bond strength and durability using the proposed technique.

Materials and Methods: Fifty-four zirconia discs were fabricated and divided into three groups ($n = 18$) according to their surface treatment: as-sintered surface (control group), airborne-particle abrasion (50-μm aluminum oxide), and SIE group. The zirconia discs were bonded to preaged composite resin discs using a light-polymerized adhesive resin (Panavia F 2.0). The zirconia/resin bond strength was evaluated using microtensile bond strength test (MTBS), and the test was repeated after each of the following intervals of accelerated artificial aging (AA): thermocycling (10,000 cycles between 5 and 55°C), 4 weeks of water storage (37°C), and finally 26 weeks of water storage (37°C). Silver nitrate nanoleakage analysis was used to assess the quality of zirconia/resin interface. A repeated measures ANOVA and Bonferroni post hoc test were used to analyze the data ($n = 18$, $\alpha = 0.05$)

Results: There were significant differences in the MTBS values between the three test groups at each of the test intervals ($p < 0.001$). AA resulted in reduction in the bond strength of the as-sintered and the particle-abraded groups (5.9 MPa and 27.4, MPa, respectively). Reduction in the bond strength of these groups was explained by the observed nanoleakage across the zirconia/resin interface. The bond strength of the SIE specimens was stable after completion of AA (51.9 MPa), which also demonstrated a good seal against silver nitrate penetration across the zirconia/resin interface.

Conclusion: SIE established a strong, stable, and durable bond to zirconia substrates. Conservative resin-bonded zirconia restorations are now possible using this new technique.

The introduction of zirconia-based materials to the dental field broadened the design and application limits of all-ceramic restorations. As a result of the unique mechanical properties of zirconia framework materials, three- or four-unit-fixed partial dentures (FPDs) are no longer the safe limit for the construction of all-ceramic restorations. Combined with CAD/CAM technology, the fabrication of extensive zirconia restorations became a simple and an accurate procedure.[1]

Chemical bonding is the basic fundamental for minimally invasive dentistry where the retention of the restoration mainly depends on adhesion to the tooth structure instead of retentive features made in the preparation.[2] Additionally, it reduces microleakage, tooth sensitivity, and the possibility of recurrent decay.[3] Patients have a preference for methods that can spare the reduction of their sound abutment teeth and would welcome adhesively bonded restorations if their retention is effective.[4,5]

Due to their chemical inertness, zirconia frameworks are resistant to aggressive chemical agents such as strong acids, alkalis, or organic and inorganic dissolving agents. Such chemical stability predicts superior long-term performance under the tough conditions of the oral environment. On the other hand, establishing a chemical bond with these materials has proved to be difficult, as they do not respond to common bonding methods used with other glass-containing ceramics, such as acid etching and silanation.[6,7] Various chemical products were developed to enhance the bond of resin cements to zirconia, that is, trialkoxysilane coupling agents,[8] but currently the only effective chemical agent to establish a relatively stable bond to zirconia substrates is the use of a phosphate ester monomer, 10-methacryloyloxydecyl dihydrogenphosphate (MDP).[9-11] Nevertheless, the established bond strength was not sufficient for retaining adhesive zirconia restorations, as debonding under function was previously reported.[7,12] Various silicoating methods were investigated[13,14] and proved inefficient in increasing the retention of zirconia crowns.[15]

Several techniques based on increasing the surface roughness, such as airborne-particle abrasion and roughening with diamond points, also failed to establish adequate mechanical retention to zirconia substrates.[7,13-17] Airborne-particle abrasion, one of the most-investigated methods, provides good bond strength to zirconia when combined with phosphate ester monomer. On the other hand, particle abrasion of zirconia results in the creation of sharp cracks and structural defects that render the zirconia framework susceptible to radial cracking during function.[18] All these bonding complications remained as obstacles against the widespread use of minimally invasive resin-bonded zirconia restorations. Nevertheless, other complicated techniques, such as fusion of glass beads and plasma spraying, gave promising results.[6]

Unfortunately, the long-term reliability of these methods could not be established based only on initial bond strength values, and long-term evaluation is required to assess bond performance under functional conditions.[19,20] As the widespread use of a resin-bonded restoration would be affected by the long-term deterioration in the properties of the established bond strength, different artificial aging (AA) techniques were employed to study their long-term performance. Water storage, thermocycling, cyclic loading, and die penetration tests were used for such purpose.[21-25]

Surprisingly, despite its reputation as a steel ceramic, zirconia is a dynamic material at an ultrastructural level. Besides its unique tetragonal monoclinic transformation, other dynamic changes occur in response to mechanical and thermal stresses.[26] Grain growth and widening of grain boundary regions could be achieved by heating zirconia in open air for few minutes at high temperatures (900 to 1250°C). Prolonged thermal etching would finally result in the formation of cubic grains, which affects the structural stability of zirconia. Additionally, it would result in structural changes of the surface of zirconia as surface lifts, increased surface roughness, and grain pull-out.[27-29] High deformation strains (up to 400%) could also be achieved by applying mechanical stresses at high temperature ranges, resulting in plastic deformation without fracturing the strained zirconia. Such properties were used in the industrial field to form complicated shapes.[30] These structural changes were related to grain sliding, splitting, and rearrangement micromovements of zirconia, which reflect the dynamic possibilities of this polycrystalline ceramic.[31]

Despite its chemical inertness, diffusion of small dopant phases, such as silica, sodium, potassium, and magnesium has been reported at grain boundary regions in several studies.[31-35] This grain boundary diffusion is directly influenced by controlled heating of zirconia to moderate temperature ranges (700 to 900°C). The presence of the dopant phases at grain boundary regions exerts high capillary and surface tension forces, which further enhance grain sliding, splitting, and rearrangement movements previously described,[31] indicating that the surface grains of zirconia could be manipulated to create a retentive surface by controlling its dynamic properties.

A new surface treatment method, selective infiltration etching (SIE), was developed for dental and biomedical applications. It transforms the surface of zirconia from a dense, nonretentive, relatively smooth, and low energy surface to a highly active and retentive surface. In this method, the surface of zirconia is coated with a glass-containing conditioning agent and heated above its glass transition temperature where grain boundary diffusion of the glass is optimized. The presence of the molten glass at grain boundaries results in sliding and splitting of the surface grains[36] and exerts surface tension and capillary forces, which overcome the grain boundary energy levels.[37] After cooling to room temperature, the glass is dissolved in an acidic bath, exposing the newly created retentive surface.[38] Zirconia resin bond strength in the range of 50 to 55 MPa was claimed to be possible using this surface treatment.[38]

In a recent study, zirconia/resin microtensile bond strength was evaluated using three surface treatment methods: SIE, airborne-particle abrasion, and coating with MDP monomer. The SIE specimens demonstrated significantly higher bond strength values than the other groups.[39] A point of concern remained, however, concerning the durability of the established bond under function and its long-term resistance to microleakage.

The aim of this research was to evaluate zirconia/resin bond strength using the new technique. Microtensile bond strength test, accelerated artificial aging, silver nitrate nanoleakage, and scanning electron microscopy (SEM) were used to evaluate the bond strength and durability. The proposed hypothesis was that the zirconia/resin bond strength could be improved using the new technique.

Materials and methods

Preparation of the specimens

Fifty-four zirconia discs (19.4-mm diameter, 3-mm thick) were prepared by cutting CAD/CAM milling blocks (Lava zirconia, 3M ESPE, St. Paul, MN). The disc-shaped specimens were then sintered in the relevant equipment and were equally divided into three groups ($n = 18$). The first group was used as a negative control (as-sintered surface), the second group was airborne-particle abraded with 50-μm aluminum oxide particles (S-U-Alustral, Schuler-Dental, Eberhard-Finckh, Germany) at 0.2 MPa pressure (P-G 400, Hornisch + Rieth, Winterback, Germany), while the third group received SIE surface treatment.

SIE technique

The bonding surface of the third group was subjected to SIE surface treatment, where the specimens were coated with a thin layer of a glass-conditioning agent composed of silica (65% wt), alumina (15% wt), sodium oxide (10% wt), potassium oxide (5% wt), and titanium oxide (5% wt). The thermal expansion coefficient of the glass closely matched that of zirconia (10.2 μm/°C) to prevent generation of possible prestresses. The composition of the glass-conditioning agent was slightly modified compared to a previous study, but the composition of silica was according to the author's guidelines.[38]

The powder of the glass was mixed with water to give a thin, creamy mixture, which was applied on the surface of the specimens using a thin brush. The specimens were then heated in open air to 650°C for 3 minutes and then cooled to 23°C. The heating and cooling rates (90°C/min) were controlled by a computer-calibrated electrical induction furnace (Austromat 3001, Dekema Dental-Keramiköfen GmbH & Co, Freilassing, Germany). After cooling to room temperature, all traces of the conditioning agent were completely washed away in 5% hydrofluoric ultrasonic bath for 15 minutes, followed by rinsing with water for 5 minutes after etching. SEM (XL 20, Philips, Eindhoven, The Netherlands) was performed after completion of the surface treatment to ensure that that the required surface topography was achieved, while element diffraction X-ray microanalysis (EDAX Inc, Mahwah, NJ) was performed to ensure complete removal of all traces of the conditioning glass.[38]

Composite resin discs (Filtek Z 250, A2, 3M ESPE) of the same previous dimensions (19.4-mm diameter, 3-mm thick) were prepared by incrementally filling a plastic mold. The discs were light-polymerized with an LED unit (Elipar, FreeLight 2, 3M ESPE). Light intensity (800 mW/cm^2) was continuously monitored (Demetron 100, Demetron Research Corp, Danbury, CT). Finally, the composite discs were postpolymerized in an intense heat and light source (Lumamate 100, Ivoclar Vivadent, Schaan, Liechtenstein), then stored in demineralized water for 3 months (T340, Heraeus GmbH, Hanau, Germany). This water storage period prevented water imbibition during artificial aging, which could result in weakening of the established bond strength.

Cementation technique

Adhesive resin cement (Panavia F 2.0, Kuraray Co., LTD, Osaka, Japan) was selected for bonding the composite discs to the zirconia substrate. For the three test groups, the zirconia disc was then coated with homogenously mixed adhesive cement (Panavia F 2.0 was stored at 6°C prior to immediate use), the composite disc was seated on top with a fixed pressure (50 N for 1 minute), and excess cement was wiped off. A specific loading device and a vertical micrometer ensured even cement thickness (30 μm) for all specimens. Finally, the adhesive cement was light-polymerized at four locations on the specimens for 60 seconds each (Elipar FreeLight 2). The cementation procedure was conducted immediately after the required surface preparation for every test group to prevent possible contamination of the specimens.

Microtensile bond strength test (MTBS)

The 18 zirconia composite resin specimens from each group were sectioned into microbars (1-mm^2 cross-section, 6-mm long) using a diamond-coated disc under water cooling (Isomet 1000; Buehler, Lake Bluff, IL). At least 25 microbars were obtained from each specimen (400 microbar/group). The bars were examined under a light microscope and only structurally intact, crack-free bars were selected (SZ, Olympus, Tokyo, Japan). Independently, 18 microbars/group (one microbar from each prepared disc-shaped specimen) were then immediately bonded to a custom-made attachment unit using a light-polymerized adhesive resin (Clearfil SE Bond, Kuraray) taking care to center the zirconia/resin interface at the free space of the attachment unit.[29] The microbars were loaded at a crosshead speed of 1mm·min^{-1} (Instron 6022, Instron Limited, High Wycombe, UK) to failure by applying an axial load to the zirconia/resin interface. The load cell (200 N) was calibrated using standardized specimens, while the crosshead speed was monitored using a computer-controlled digital micrometer (Millitron, Feinpruf Perthen GmbH, Gottingen, Germany). The broken microbars were examined under SEM to assess the fracture pattern. These data were used as an indication of the initially established zirconia/resin bond strength for each group.[40]

AA technique

The remaining zirconia composite resin microbars (382/group) were thermocycled in a water bath (10,000 cycles between 5 and 55°C and a dwell time of 3 minutes); then the MTBS test was carried out again after completion of the thermocycling program, after 4 weeks, and finally after 26 weeks, during which the bars were stored in demineralized water at 37°C (18 microbars/group/test interval).

Assessment of nanoleakage

Nanoleakage technique depends on immersion of bonded interfaces in silver nitrate solution where nano-silver particles could diffuse and penetrate along nano-gaps and structural defects. After diffusion, the silver particles are fixed using x-ray developing solution and detected using SEM. After completion of AA (26 weeks), three zirconia composite resin specimens from each test group were immersed in a buffered solution of silver nitrate (6 hours). The specimens were then washed under running water, fixed in X-ray developing liquid under intense light (10 hours), washed, and dried. Finally the specimens were sliced into small sections, ultrasonically cleaned (Sonorex RK 102, Bardelin electronic KG, Berlin, Germany), dried, gold sputter coated (S150B sputter coater, Edwards, Crawly, UK), and examined under SEM. The zirconia/resin interface was examined at high magnifications, and silver nitrate particles were identified microscopically and confirmed using EDAX analysis.

Statistical analysis

Repeated measures ANOVA was selected to analyze the data with one within-subject factor (time, four levels) and one between-subjects factor (zirconia surface, three levels). Bonferroni post hoc test was selected for pairwise comparisons ($\alpha = 0.05$). The sample size (n = 18 discs/group/test interval) was based on a power analysis study (power = 1) set to detect medium effect size differences (F = 0.25), which in terms of MTBS values, could result in clinically justified recommendations. Data were digitally examined and analyzed (SPSS 14.0; SPSS, Inc, Chicago, IL).

Results

The reliability of the MTBS as a bond-strength-measuring method was evaluated using interclass correlation coefficient

Table 1 Microtensile bond strength values (MPa) and fracture pattern at different test intervals

Zirconia surface	Initial value	After thermocycling	After 4 weeks of water storage	After 26 weeks of water storage
As-sintered	22.6 (8.2)^{a} (50% interfacial)	7.9 (7.4)^{b,c} (100% interfacial)	6.9 (6.4)^{b,c} (100% interfacial)	5.9 (6.5)^{c} (100% interfacial)
Airborne-particle abrasion	35.9 (5.1)^{a} (80% cohesive)	34.7 (3.3)^{a,b} (50% cohesive)	28.7 (7.3)^{a,b} (65% interfacial)	27.4 (3.4)^{b} (80% interfacial)
Selective infiltration etching	51.9 (4.7)^{a} (95% cohesive)	53.4 (5.8)^{a} (95% cohesive)	52.5 (3.9)^{a} (90% cohesive)	51.9 (4.5)^{a} (80% cohesive)

Small letters within a group indicate no significant statistical difference in MTBS values between different test intervals.
There were significant statistical differences in MTBS values between the three tested groups at the same test interval.

with average measures = 0.99, indicating that the MTBS values made from each of the 18 discs/group at one time interval were statistically comparable. Such data indicated that the tested microbars were independent from their preparation source (the bonded disc specimens). Adjusted partial eta squared ($\eta = 0.99$) demonstrated the precision of MTBS as a test of choice, which resulted in a low percentage of variance.

There were significant statistical differences in the MTBS values between the three groups (F = 906.6, $p < 0.001$), between the different AA intervals (F = 13.8, $p < 0.001$) and their interaction (F = 5.4, $p < 0.001$). For the same test interval, all MTBS values between the different groups were significantly different.

AA had a significant deteriorating effect on the bond strength of the as-sintered and airborne-particle abrasion groups as demonstrated by significant reduction in their bond strength values after thermocycling and water storage. The initial MTBS values significantly decreased after completion of AA from 22.65 MPa to 5.9 MPa (as-sintered control) and from 35.9 MPa to 27.4 MPa (airborne-particle abrasion) except for the SIE group, where the initial bond strength value of 51.9 MPa was maintained after completion of AA (Table 1).

SEM examination of the initial surface of the zirconia discs (as-sintered surface) revealed densely packed zirconia grains (Fig 1A). On the other hand, airborne-particle abrasion resulted in increasing the surface roughness of zirconia and in the creation of sharp cracks and surface scratches (Fig 1B). After completion of SIE, the surface of zirconia demonstrated intergrain porosities as a result of grain splitting, sliding, and microrearrangement movements influenced by the glass infiltration and temperature (Fig 1C). SEM analysis of the failed microbars revealed a predominantly cohesive fracture pattern for the SIE group, which did not change during the AA process, while interfacial failure was mainly observed for the other two groups, which increased during successive testing intervals (Table 1).

Nanoleakage assessment revealed silver nitrate penetration the across zirconia/resin interface observed for the as-sintered and the particle-abraded groups (Fig 2A and B). The SIE group demonstrated infiltration of the adhesive resin into the created microporosities on the surface of zirconia as result of SIE, which prevented nanoleakage (Fig 2C).

Discussion

The adoption of MTBS test for measuring bond strength has many advantages over other testing methods like shear bond strength,[8,41,42] as the applied tensile stresses are vertical to the bonded area.[43] Moreover, the small cross-section of the microbars ensures less incorporation of structural defects, resulting in a low percentage of variance ($\eta = 0.99$) of the data. On the other hand, it is a very tedious test, which requires investing much time and effort, especially during cutting the microbars, to avoid accidental damage to the specimens.

The results of this study indicated that MTBS values in the range of 22 to 36 MPa could be established using MDP-containing adhesive resin in combination with either as-sintered or particle-abraded surfaces, but the bond is susceptible to hydrolysis during function, as reduction in bond strength values was observed during the AA program for both groups. The rapid reduction in bond strength observed for the as-sintered group was previously reported in other studies[9,23] but the observed reduction for the particle-abraded group in combination with application of MDP monomer is in direct disagreement with their findings, which reported bond stability for up to 2 years of water storage. Such contradiction could be related to the fact that these studies used Plexiglas tubes, which could isolate the bonded interface from the influence of water. Moreover, using microbars instead of discs exposes more surface area of the bonded interface to the influence of water.

Regarding the particle-abraded group, SEM examination of the zirconia/resin interface revealed the presence of microgaps and structural defects, which were a source for silver nitrate nanoleakage due to deterioration of the bond between the resin cement and the substrate (Fig 2B). Thus, the role the MDP monomer plays in providing a durable chemical bond to zirconia seems questionable, in contradiction to previous studies.[9,10] One should consider that this study used microbars, which exposed a larger surface area of the bonded interface to the influence of thermocycling and water storage compared to specimens prepared for shear test, where only the periphery of the bonded interface is exposed to the effect of AA.

Additionally, omitting particle abrasion as a pretreatment resulted in complete gap formation, intense nanoleakage, and premature failure, which also minimizes the role of the MDP agent and draws attention to the mechanical retention achieved by particle abrasion (Fig 2A). These observations explain the reduction in MTBS values and the increase in the percentage of interfacial failure during successive AA test intervals observed for the as-sintered and airborne-particle abraded groups.[44,45]

The surface topography created by the SIE technique created a highly retentive surface where the adhesive resin was directly able to penetrate and interlock, resulting in significantly higher bond strength. The proposed hypothesis was thus

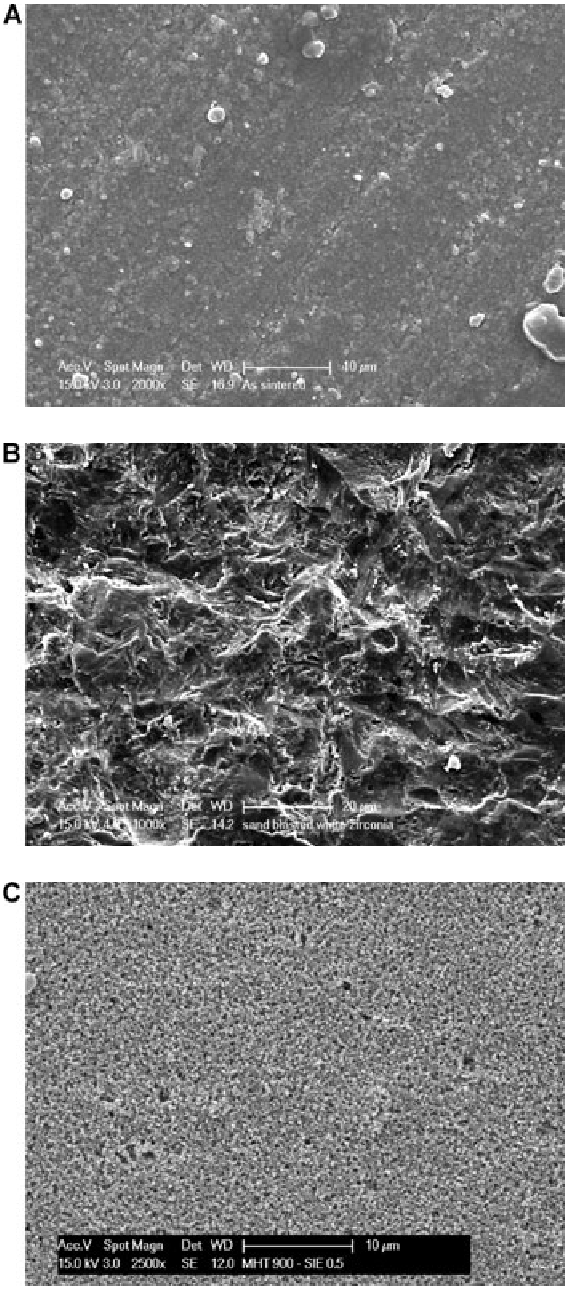

Figure 1 (A) Scanning electron microscopy (SEM) image (2000×) demonstrating the dense nonretentive surface of as-sintered zirconia specimens. (B) SEM image (1000×) demonstrating the surface of zirconia after airborne- particle abrasion, which resulted in the creation of sharp cracks and surface defects. (C) SEM image (2500×) of a zirconia specimen demonstrating the created retentive surface architecture after completion of selective infiltration etching (SIE). Grain sliding and splitting resulted in the creation of intergrain porosities and nano-spaces where the adhesive resin can infiltrate.

accepted. Even though the depth of resin tag penetrations was limited to 0.3 to 0.7 μm, which could be altered by changing the SIE protocol, the architecture, direction, and orientation of the created nano-retentive intergrain porosities allowed for the

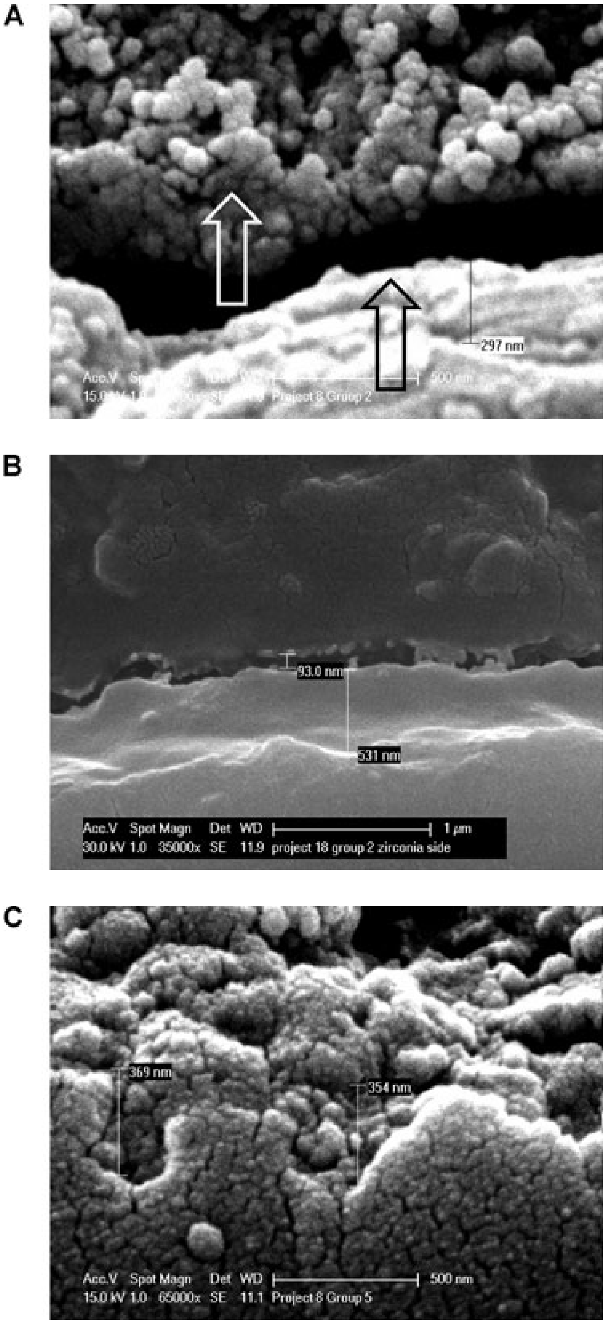

Figure 2 (A) SEM image (50,000×) showing deterioration of the zirconia/resin bond after artificial aging, which resulted in microgaps and intense silver nitrate nanoleakage (as-sintered group). Silver nitrate nanoparticles (white arrow) appear as gray spheres above the observed nanogap (black arrow). (B) SEM image (35,000×) demonstrating silver nitrate nanoleakage across the zirconia/resin interface as a result of disruption of the bond between the resin cement and the airborne-particle-abraded zirconia. (C) SEM image (65,000×) demonstrating the resin cement (upper half) filling the nano-porosities created on the surface of zirconia (lower half) as a result of SIE. No microgaps or nanoleakage were observed after artificial aging. Notice that the depth of the created surface porosities was limited to 0.36 μm, while the bulk of zirconia was not affected.

establishment of a strong nano-mechanical bond with the adhesive resin used. In addition to the high bond strength value, the new surface treatment resulted in the creation of a sealed interface, which resisted nanoleakage during AA (Fig 2C).

A point of concern was the effect of the created inter-grain porosities on the mechanical properties of the SIE zirconia. In a previous study performed by the authors during technique development, cross-section studies combined with ultrastructural analysis revealed that the depth of the created porosities did not exceed 1.5 μm. This suggests that the proposed treatment surface porosities involved only the outer 5 to 6 grains, as the average grain size of zirconia is 0.3 μm (Fig 2C). Additionally, 4-point flexural strength of SIE zirconia was comparable to the particle-abraded specimens, as the created surface architecture occurs on a nano-metric level.[39] On the other hand, airborne-particle abrasion resulted in excessive surface roughness ($R_a = 6$ to 9 μm), material loss, and sharp cracks (Fig 1B).[46] Regarding the subsurface cracks observed in some SIE specimens, it should be emphasized that the prepared specimens had a relatively large bonding surface area, which is much larger than the range of practical applications, and such a finding would be unexpected during zirconia restoration bonding.[47] More studies should be conducted on this new technique to reveal its influence on the restoration.

The adhesive resin fills a complex interface, and establishment of a reliable bond to zirconia was proven to be difficult using traditional methods. SIE produced a strong, durable, and stable zirconia/resin bond strength, which resisted nanoleakage during AA. Minimally invasive resin-bonded zirconia FPDs are now possible using this new surface treatment.

Conclusions

Within the limitations of this study, zirconia/resin bond strength could be significantly improved using SIE technique in combination with MDP monomer.

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