Effect of etching and airborne particle abrasion on the microstructure of different dental ceramics

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Statement of problem. The ceramic composition and microstructure surface of all-ceramic restorations are important components of an effective bonding substrate. Both hydrofluoric acid etching and airborne aluminum oxide particle abrasion produce irregular surfaces necessary for micromechanical bonding. Although surface treatments of feldspathic and leucite porcelains have been studied previously, the high alumina-containing and lithium disilicate ceramics have not been fully investigated.

Purpose. The purpose of this study was to assess the surface topography of 6 different ceramics after treatment with either hydrofluoric acid etching or airborne aluminum oxide particle abrasion.

Material and methods. Five copings each of IPS Empress, IPS Empress 2 (0.8 mm thick), Cergogold (0.7 mm thick), In-Ceram Alumina, In-Ceram Zirconia, and Procera (0.8 mm thick) were fabricated following the manufacturer's instructions. Each coping was longitudinally sectioned into 4 equal parts by a diamond disk. The resulting sections were then randomly divided into 3 groups depending on subsequent surface treatments: Group 1, specimens without additional surface treatments, as received from the laboratory (control); Group 2, specimens treated by use of airborne particle abrasion with 50-μm aluminum oxide; and Group 3, specimens treated with 10% hydrofluoric acid etching (20 seconds for IPS Empress 2; 60 seconds for IPS Empress and Cergogold; and 2 minutes for In-Ceram Alumina, In-Ceram Zirconia, and Procera).

Results. Airborne particle abrasion changed the morphologic surface of IPS Empress, IPS Empress 2, and Cergogold ceramics. The surface topography of these ceramics exhibited shallow irregularities not evident in the control group. For Procera, the 50-μm aluminum oxide airborne particle abrasion produced a flattened surface. Airborne particle abrasion of In-Ceram Alumina and In-Ceram Zirconia did not change the morphologic characteristics and the same shallows pits found in the control group remained. For IPS Empress 2, 10% hydrofluoric acid etching produced elongated crystals scattered with shallow irregularities. For IPS Empress and Cergogold, the morphologic characteristic was honeycomb-like on the ceramic surface. The surface treatment of In-Ceram Alumina, In-Ceram Zirconia, and Procera did not change their superficial structure.

Conclusion. Hydrofluoric acid etching and airborne particle abrasion with 50-μm aluminum oxide increased the irregularities on the surface of IPS Empress, IPS Empress 2, and Cergogold ceramics. Similar treatment of In-Ceram Alumina, In-Ceram Zirconia, and Procera did not change their morphologic microstructure. (J Prosthet Dent 2003;89:479-88.)

CLINICAL IMPLICATIONS

Hydrofluoric acid etching and airborne aluminum oxide particle abrasion did not change the surface microstructure of In-Ceram Alumina, In-Ceram Zirconia, and Procera ceramics. Alternate protocols are recommended to ensure a proper surface for adequate bonding.

Dental ceramics are appreciated as highly esthetic restorative materials with optimal esthetic properties that better simulate the appearance of natural dentition. Other desirable characteristics include translucence, fluorescence, chemical stability, biocompatibility, high compressive strength, and a coefficient of thermal expansion similar to that of tooth structure. In spite of their many advantages, ceramics are fragile under tensile

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Table I. Description of ceramic materials used in study

Ceramic	Manufacturer	Composition*
IPS Empress	Ivoclar-Vivadent, Schaan, Liechtenstein	SiO_2 (63%), Al_2O_3 (17.7%), K_2O (11.2%), Na_2O (4.6%), CeO_2 (1.6%), B_2O_3, CaO, BaO, TiO_2 (< 1%)
IPS Empress 2	Ivoclar-Vivadent, Schaan, Liechtenstein	SiO_2 (57 - 80%), Al_2O_3 (0 - 5%), La_2O_3 (0.1 - 6%), MgO (0 - 5%), ZnO (0 - 8%), K_2O (0 - 13%), Li_2O (11 - 19%), P_2O_5 (0 - 11%)
Cergogold	Degussa Dental, Hanau, Germany	SiO_2, Al_2O_2, K_2O, Na_2O, CaO
In-Ceram Alumina	Vita Zahnfabrik, Seefeld, Germany	Al_2O_3 (82%), La_2O_3 (12%), SiO_2 (4.5%), CaO (0.8%), other oxides (0.7%)
In-Ceram-Zirconia	Vita Zahnfabrik, Seefeld, Germany	Al_2O_3 (62%), ZnO (20%), La_2O_3 (12%), SiO_2 (4.5%), CaO (0.8%), other oxides (0.7%)
Procera AllCeram	Nobel Biocare, Göteborg, Sweden	Al_2O_3 (99.5%)

*According to the manufacturer.

strain.[1-3] This weakness can be attributed to the presence and propagation of microflaws present on the surface of the material, making the ceramic susceptible to fracture during the luting procedure and under occlusal force.[1-3] The ceramo-metal restoration, which combines the strength of metal with the esthetics of ceramic, improved the success of dental ceramics.[4,5] The ceramo-metal restorations enjoy wide clinical use; however, the metal core can reduce the translucency of the restoration.

McLean and Hughes[1] developed the first strengthened ceramic in 1965 by changing the composition of the crystalline phase with the addition of aluminum crystals. Subsequently, the introduction of ceramics with different compositions combined with the use of novel laboratory techniques has resulted in improved mechanical properties and heightened esthetics of these restorations.[6] These strengthened all-ceramic restorations have been indicated for inlays, onlays, crowns, and fixed partial dentures.[7]

The cementation process is vital for the clinical success of all-ceramic restorations. The restoration may be cemented with zinc phosphate, glass ionomer, or composite cements. The success of the cementation process is dependent on the composition of the ceramic material. When zinc phosphate or glass ionomer cements are used, mechanical retention is necessary. Such water-based cements work mainly by frictional force. On the other hand, when mechanical retention is compromised, adhesive luting systems are recommended. The bond of the resin luting cement to the tooth structure is enhanced by acid etching of enamel or dentin and by the use of a dentin adhesive. The penetration of monomers into the demineralized dentinal matrix, followed by polymerization, promotes the micromechanical bond via hybrid layer formation.[8-10] In a similar way, the internal surface of the ceramic restoration must be prepared to optimize the micromechanical bond between the ceramic and the resin. To prepare the feldspathic ceramic surface for bonding, hydrofluoric acid etching is recommended. The microstructure of this ceramic changes by dissolution of one of the glassy phases of porcelain.[11,12] This phase is dissolved preferentially to create an appropriate microstructure for bonding.[13-15] Another prebonding treatment recommended for ceramic surfaces is airborne aluminum oxide particle abrasion.[16,17] Because the ceramic has components bondable to silane, the cementation process may also be enhanced by application of a silane coupling agent. These agents are capable of forming chemical bonds between the inorganic phase of the ceramic and the organic phase of the resin.[18-23]

Previous studies have shown that the all-ceramic restorations based in densely sintered high-purity alumina and glass infiltrated aluminum oxide resist forming microretentive surfaces after hydrofluoric acid etching and airborne particle abrasion surface treatment.[24,25] Scanning electron microscopy (SEM) revealed only an irregular surface texture. The shear bond strength of resin luting agents to ceramics with high aluminum content was lower for specimens prepared by hydrofluoric acid etching than for those prepared by airborne particle abrasion.[25,26]

The aim of this study was to assess the surface topography of 6 different ceramics after hydrofluoric acid etching or by airborne aluminum oxide particle abrasion treatments. The differences in composition and microstructure of all-ceramic restorations might be an important factor in obtaining effective bond strengths between the ceramic and resin luting agent.

MATERIAL AND METHODS

All specimens in this study were fabricated following the manufacturer's instructions. Five copings each of IPS Empress, IPS Empress 2, Cergogold, In-Ceram Alumina, In-Ceram Zirconia, and Procera were fabricated on a die stone master cast. Compositions of the ceramics are listed in Table I.

Specimen fabrication

For IPS Empress specimens, 2 layers of die spacer were applied over the stone die and wax copings, 0.8

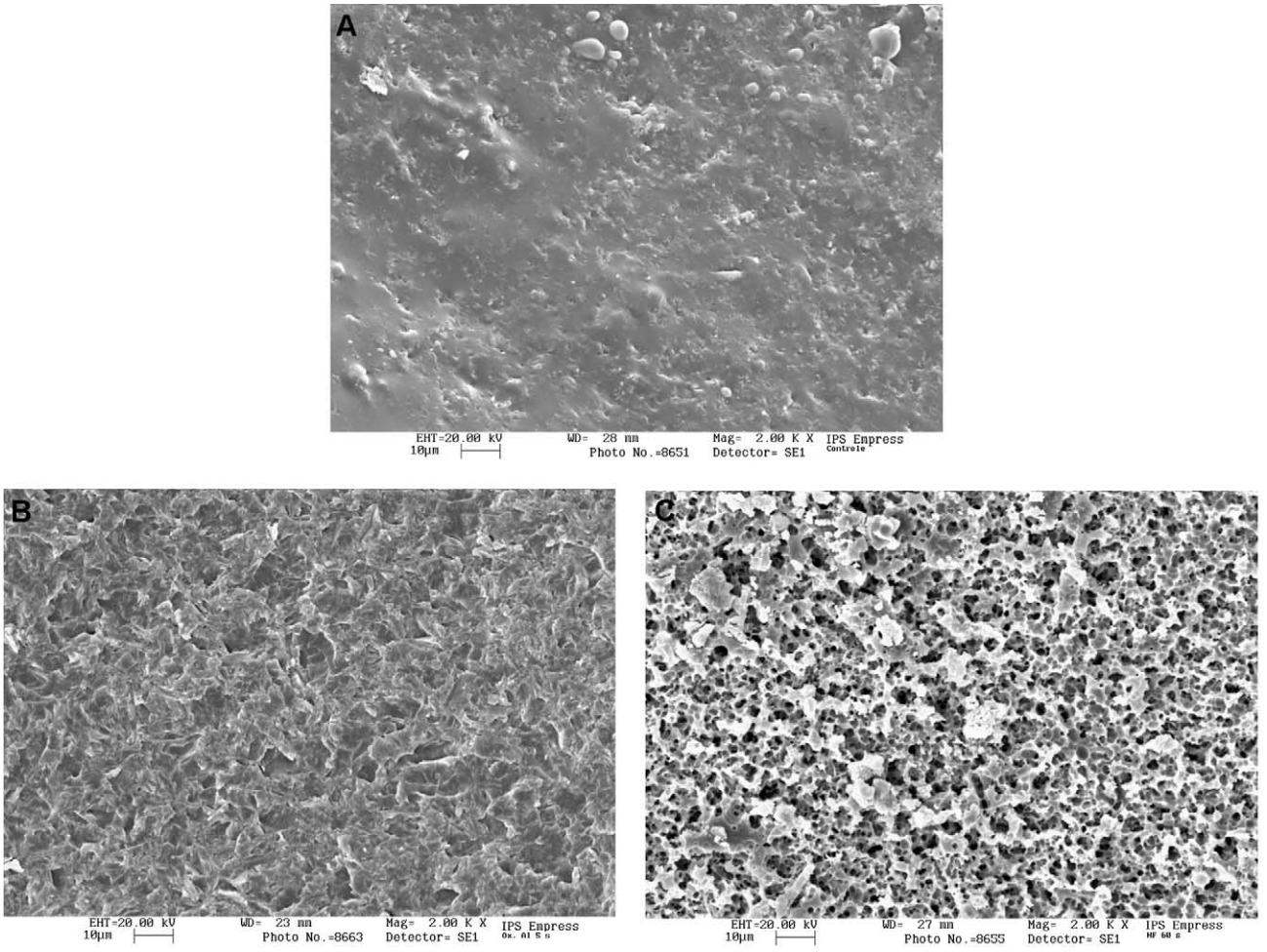

Fig. 1. SEM of IPS Empress. **A**, Control. (Original magnification ×2000.) **B**, Airborne particle abrasion with 50-µm aluminum oxide for 5 seconds. (Original magnification ×2000.) **C**, Etching with 10% hydrofluoric acid for 60 seconds. (Original magnification ×2000.)

mm thick, were prepared. The wax patterns were invested in IPS Empress investment and eliminated in a burnout furnace (7000-5P; EDG Equipments Ltda, São Carlos, Brazil) by heating the refractory die at the same time the IPS Empress ingots (color A2) and the alumina plunger were heated at 3°C per minute to 850°C and held for 90 minutes. After the procedure described, the investment, plunger, and ingot were transferred to a furnace (EP 500; Ivoclar-Vivadent, Schaan, Liechtenstein) that increased the temperature to 1180°C and automatically pressed the melted ingot to the mold. After pressing and cooling to room temperature, the specimens were divested with 50-µm glass beads at 2-bar pressure, ultrasonically cleaned in a special liquid (Invex liquid; Ivoclar-Vivadent) for 10 minutes, washed in running water, and dried. They were then treated with airborne particle abrasion with 100-µm aluminum oxide at 1-bar pressure.

The IPS Empress 2 wax patterns were prepared as previously described and invested in IPS Empress 2 Speed investment. The wax was eliminated in a burnout furnace pre-heated to 850°C with the alumina plunger for 90 minutes. The IPS Empress 2 ingots (color A2) became softened at 920°C and were automatically pressed into the mold in a furnace (EP 500; Ivoclar-Vivadent). After pressing and cooling to room temperature, the specimens were divested, cleaned, washed, dried, and airborne particle–abraded as described for the IPS specimens.

For Cergogold specimens, the spacer (Isolit; Degussa Dental, Hanau, Germany) was applied over the stone die and the 0.7-mm–thick wax coping was fabricated. The wax coping was invested (Cergofit investment; Degussa Dental) and allowed to set. It was then placed in a burnout furnace to eliminate the wax. The burnout furnace was preheated to 270°C, and the temperature

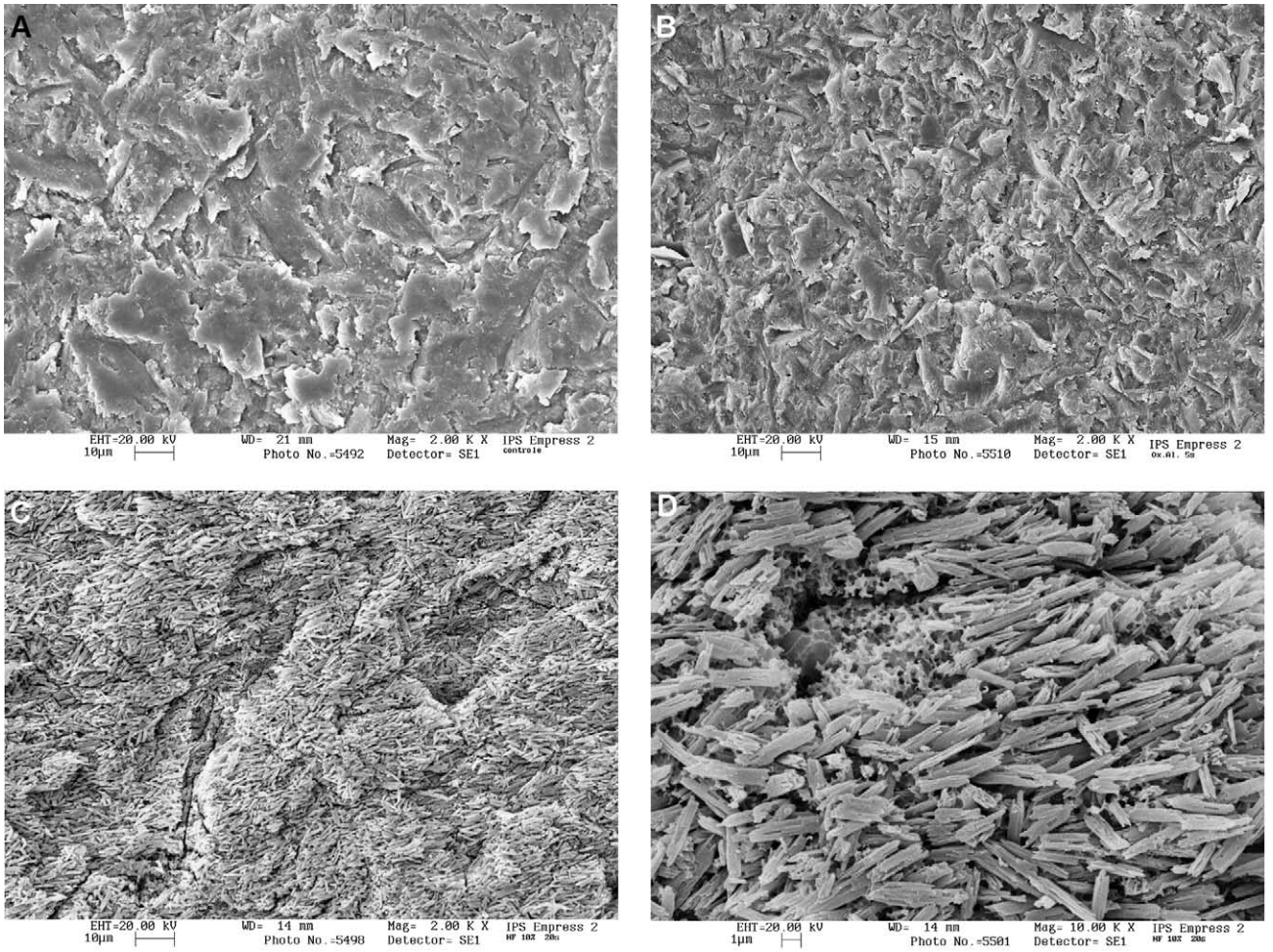

Fig. 2. SEM of IPS Empress 2. **A**, Control. (Original magnification ×2000.) **B**, Airborne particle abrasion with 50-µm aluminum oxide for 5 seconds. (Original magnification ×2000.) **C**, Etching with 10% hydrofluoric acid for 20 seconds. (Original magnification ×2000.) **D**, High magnification of etching with 10% hydrofluoric acid for 20 seconds. (Original magnification ×10000.)

gradually increased to approximately 850°C with the alumina plunger for 40 minutes. The Cergogold ingots (color A3) were pressed in an automatic press furnace (Cerampres Qex; Ney Dental Inc, Bloomfield, Conn.). After cooling, the specimens were divested using 50-µm glass beads at 4-bar pressure, followed by airborne particle abrasion with 100-µm aluminum oxide at 2-bar pressure, to remove the refractory material. Finally, the specimens were treated with airborne particle abrasion with 100-µm aluminum oxide at 1-bar pressure.

For In-Ceram Alumina and In-Ceram Zirconia specimens 3 layers of die spacer (Interspace Varnish; Vita Zahnfabrik, Bad Säckingen, Germany) were applied over the stone die. An impression was made with impression material (Vita Duplication Material; Vita Zahnfabrik), and then duplicated in a plaster (Special plaster; Vita Zahnfabrik). The aluminum oxide powder or aluminum zirconia powder were mixed with a special liquid as instructed by the manufacturer (Vita In-Ceram Alumina/Zirconia mixing liquid; Vita Zahnfabrik), and ultrasonicated (Vitasonic II; Vita Zahnfabrik) for 7 minutes. The slurry mixture was then painted over the special plaster die and fired at 1120°C in the oven (Inceramat II; Vita Zahnfabrik) for 10 hours. The sintered substructure was subsequently reduced to a thickness of 0.5 mm. Glass infiltration was obtained by coating the aluminum oxide framework with a glass powder (silicate-aluminum-lantanium)-distilled water mixture and firing in the furnace for 4 hours at 1100°C. The excess glass was removed by use of a fine-grained diamond (Renfert, Hilzingen, Germany). Finally, the coping was airborne particle–abraded with 80-µm aluminum oxide at a pressure of 3-bar. The Procera die was mounted on a rotating platform in the Procera scanner attached to a personal computer and modem. After digitization, the coping was designed on the personal computer to be 0.6

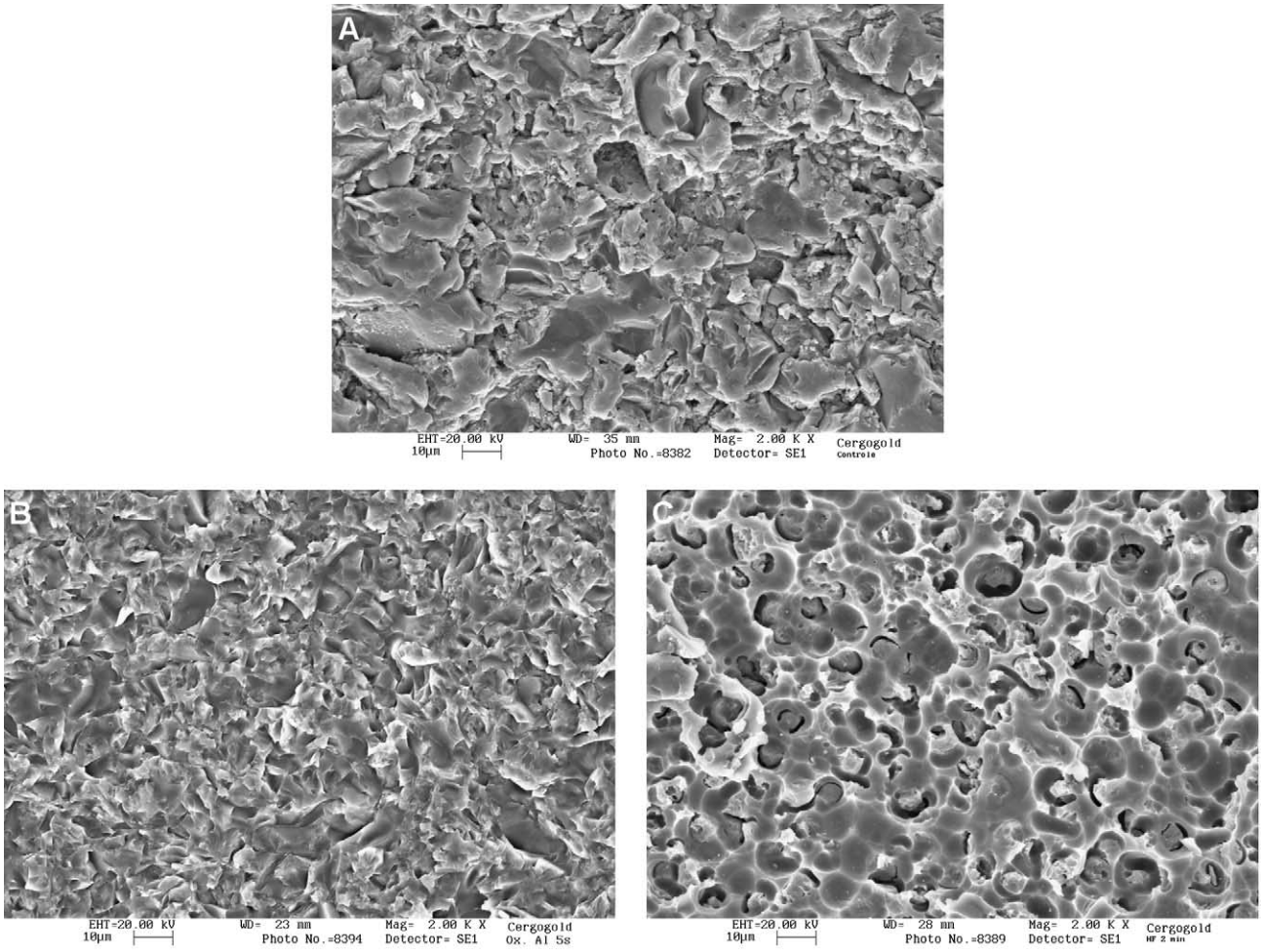

Fig. 3. SEM of Cergogold. **A**, Control. (Original magnification ×2000.) **B,** Airborne particle abrasion with 50-µm aluminum oxide for 5 seconds. (Original magnification ×2000.) **C,** Etching with 10% of hydrofluoric acid for 60 seconds. (Original magnification ×2000.)

mm thick, and then the design was transmitted by modem to the "hub" laboratory in Gothenburg, Sweden, where the coping was manufactured with the CAD/CAM technique. The data file was received by a computer-controlled milling machine, which created a duplicate of the preparation onto which aluminum oxide was densely packed. The aluminum oxide was machined to the proportions requested in the digital prescription and sintered to full density (99.5% aluminum oxide). The coping was returned for evaluation.

Surface treatment

Each coping was longitudinally sectioned in 4 equal parts with a diamond disk under water coolant. They were then randomly divided in 3 groups for different surface treatments: Group 1, specimens without additional surface treatment (controls); Group 2, specimens treated with airborne particle abrasion with 50-µm aluminum oxide for 5 seconds at 4-bar pressure. The distance of the tip from the ceramic surface was approximately 10 mm.[11,12] These specimens were washed with tap water for 1 minute, ultrasonically cleaned in a water bath for 10 minutes, and air-dried; Group 3, specimens treated with 10% hydrofluoric acid etching (20 seconds for IPS Empress 2, 60 seconds for IPS Empress and Cergogold, and 2 minutes for In-Ceram Alumina, In-Ceram Zirconia, and Procera).[11,23] After etching, the specimens were washed with tap water for 1 minute, ultrasonically cleaned in water bath for 10 minutes, and air dried.

SEM analysis

All specimens were gold coated with a sputter coater (Balzers-SCD 050; Balzers Union Aktiengeselischaft Fürstentun, Liechtentein) for 180 seconds at 40 mA. They were then mounted on coded brass stubs and examined using electron microscopy (LEO 435 VP; Cambridge, England) operated at 20 Kv, by the same operator.

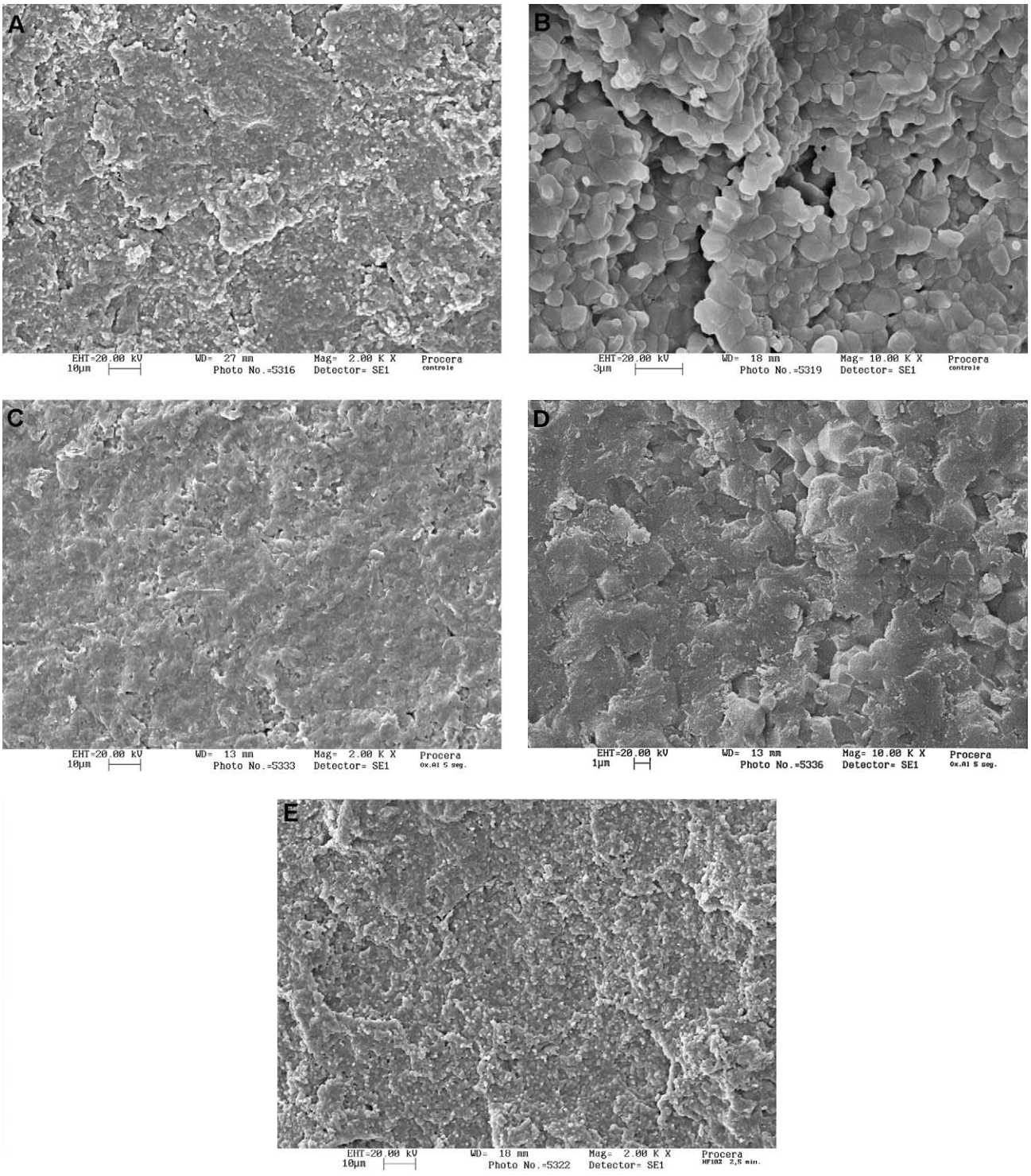

Fig. 4. SEM of Procera. **A**, Control. (Original magnification ×2000.) **B**, High magnification of control. (Original magnification ×10000.) **C,** Airborne particle abrasion with 50-μm aluminum oxide for 5 seconds. (Original magnification ×2000.) **D**, High magnification of airborne particle abrasion with 50-μm aluminum oxide for 5 seconds. (Original magnification ×10000.) **E**, Etching with 10% hydrofluoric acid for 2 minutes. (Original magnification ×2000.)

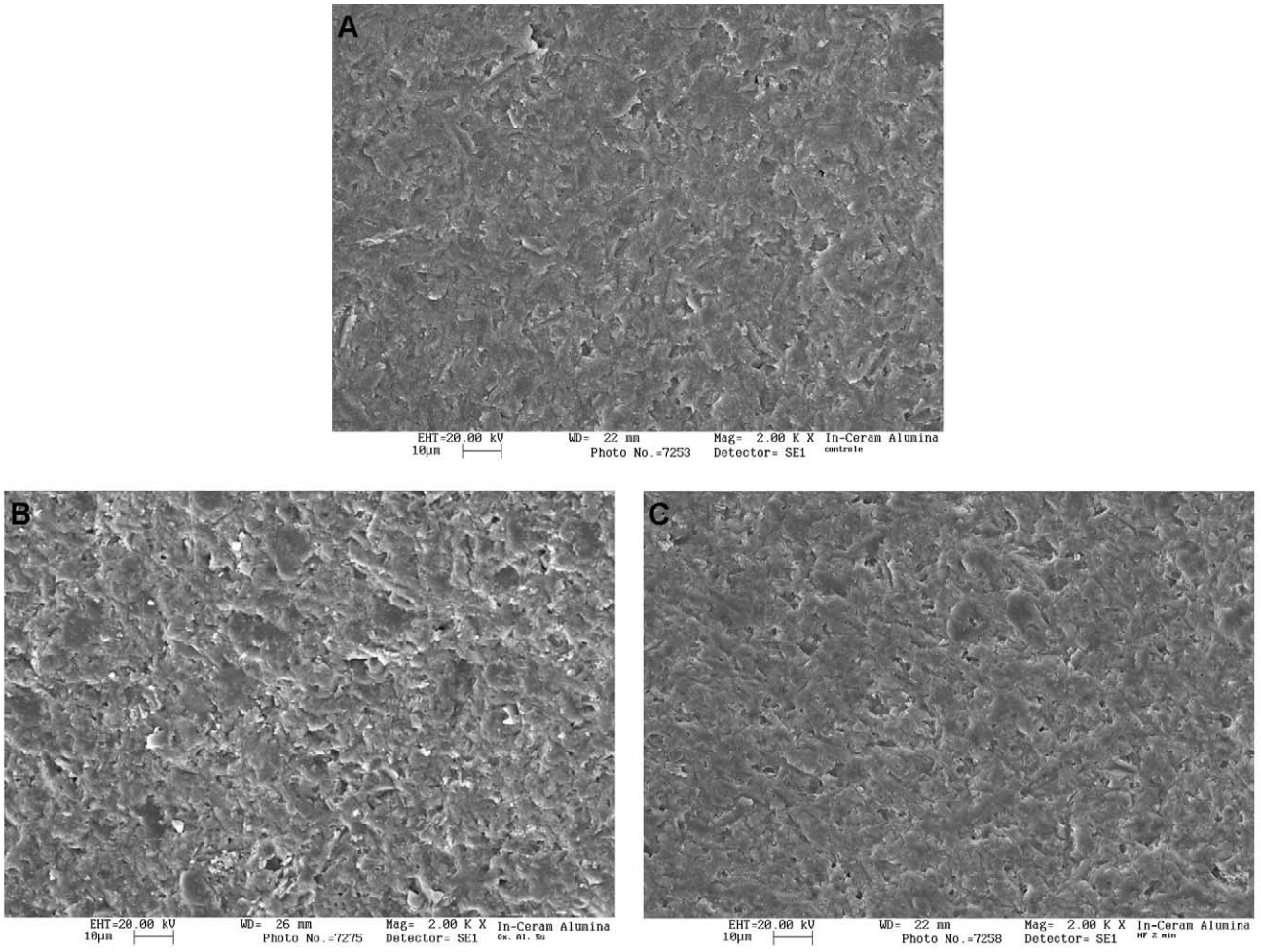

Fig. 5. SEM of In-Ceram. **A**, Control. (Original magnification ×2000.) **B**, Airborne particle abrasion with 50-µm aluminum oxide for 5 seconds. (Original magnification ×2000.) **C**, Etching with 10% hydrofluoric acid for 2 minutes. (Original magnification ×2000.)

RESULTS

The SEM photographs showed that 50-µm aluminum oxide airborne particle abrasion modified the morphologic surface of IPS Empress, IPS Empress 2, and Cergogold ceramics. The resulting surface topographies were similar between the groups. Superficial shallow irregularities similar to the control group are clearly evident (Figs. 1 *A* and *B*; 2, *A* and *B*; and 3, *A* and *B*). The Procera specimens (Fig. 4, *B*) showed 1 area with grain pullout, and the shapes of the grains of densely sintered alumina were easily identifiable. When 50-µm aluminum oxide was used for airborne particle abrasion, all grains were blunted producing a flattened surface (Fig. 4, *C* and *D*). Fifty-µm aluminum oxide airborne particle abrasion did not change the morphologic characteristics of In-Ceram Alumina and In-Ceram Zirconia, and the same surface irregularities found in the control group, shallow pits, remained (Figs. 5 *A* and *B*; and 6, *A* and *B*).

Ten percent hydrofluoric acid etching of IPS Empress 2 produced elongated crystals with shallow irregularities (Fig. 2, *C* and *D*). For the IPS Empress and Cergogold ceramics, hydrofluoric acid etching produced morphological honeycomb-like surfaces (Figs. 1, *C*; and 3, *C*). Hydrofluoric etching of In-Ceram Alumina, In-Ceram Zirconia, and Procera did not change their superficial structure when compared with the control group (Figs. 4, *A* and *E*; 5, *A* and *C*; and 6, *A* and *C*).

DISCUSSION

The micromechanical retention of the ceramic surface is very important for bonding with a resin luting cement. The unfilled resin and the resin luting cement are applied to the treated ceramic surface. This penetration and in situ polymerization is responsible for the bonding of the resin luting agent to the ceramic resto-

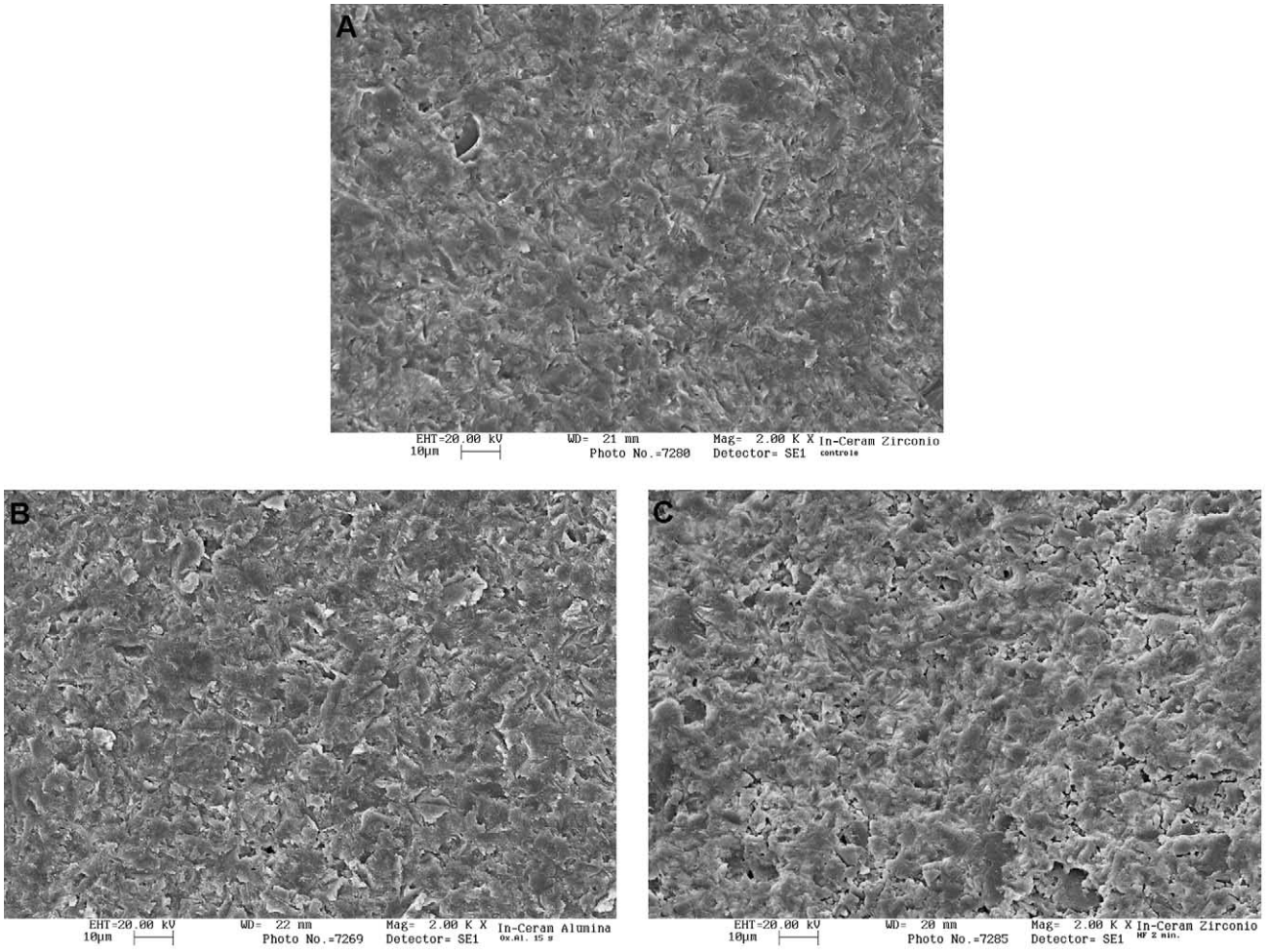

Fig. 6. SEM of In-Ceram Zirconia. **A**, Control. (Original magnification ×2000.) **B**, Airborne particle abrasion with 50-µm aluminum oxide for 5 seconds. (Original magnification ×2000.) **C**, Etching with 10% hydrofluoric acid for 2 minutes. (Original magnification ×2000.)

ration.[13,22,25,26] Resin luting agents are dependent on micromechanical retention for bonding.

Ten percent hydrofluoric acid etching changed the morphologic surface of IPS Empress and Cergogold ceramics, creating topography similar to a honeycomb (Figs. 1, *C*; and 3, *C*). The chemical etching process can be explained by the preferential reaction of the hydrofluoric acid with the silica phase of the feldspathic ceramic to form hexafluorosilicates.[11,23] These silicates are removed by rinsing with water. The final result is a honeycomb-like surface, ideal for micromechanical retention.[23]

When the ceramic surface of IPS Empress 2 was treated with hydrofluoric acid, elongated crystals and shallow irregularities were clearly observed (Fig. 2, *C* and *D*). According to Höland et al[15] the main crystal phase of IPS Empress 2 glass ceramic is formed by elongated crystals of lithium disilicate. A second phase is composed of lithium orthophosphate. A glass matrix surrounds both crystalline phases. Hydrofluoric acid is able to remove the glass matrix and the second crystalline phase thus creating irregularities within the lithium disilicate crystals. The present study shows that 10% hydrofluoric acid etching applied for 20 seconds on the IPS Empress 2 ceramic is effective in the removal of the second crystalline phase and the glass matrix and therefore creates an irregular surface suitable for bonding.

Hydrofluoric acid etching did not change the surface structure of In-Ceram Alumina and In-Ceram Zirconia ceramics. The shallow irregularities observed in the control group were also found after hydrofluoric acid etching of these ceramics. Alumina (Al_2O_3) represents 85% by weight of In Ceram Alumina and 67% by weight of In-Ceram Zirconia. Both structures are infiltrated by lantanium-aluminium-silicate glass containing less than 5% of silica by weight. As the silica phase is the only phase able to be etched by hydrofluoric acid, the etching was therefore inefficient, as shown in Figures 5, *C*, and

6, *C*. On the other hand, Procera is a ceramic with high alumina content that does not contain a glassy phase. The ceramic surface of Procera was also unetched by hydrofluoric acid (Fig. 4, *E*).

Dental laboratories customarily use 100-μm aluminum oxide particles to remove the refractory investment. This promotes morphologic alteration of the ceramic surface, resulting in an increase in the number of potential retention areas. In the present study, after the laboratory procedures, additional airborne particle abrasion was performed with 50-μm aluminum oxide particles at 4-bar pressure for 5 seconds. For IPS Empress, IPS Empress 2, and Cergogold ceramics, this treatment changed the surface by increasing the number of pits per unit area compared with the control treatment.

For In-Ceram Alumina and In-Ceram Zirconia prepared according to the manufacturer's recommendations, the irregularities appeared shallower than the control surface of other ceramics included in this study. This may be related to the high content of alumina present in these ceramics and the glass infiltrated into the framework. Therefore aluminum oxide crystals used for airborne particle abrasion have a hardness similar to that of the aluminum oxide crystals present in the ceramic structure, confirming previous studies[24,26] (Figs. 5, *A* and *B*; and 6, *A* and *B*). As an alternative surface treatment, Sen et al[17] showed that airborne particle abrasion with synthetic diamond particles of 1- to 3- μm increased the surface roughness of In-Ceram Alumina and promoted higher bond strength to the resin luting agent in comparison to airborne particle abrasion with 50-μm aluminum oxide. The airborne particle abrasion of Procera with 50-μm aluminum oxide caused flattening of the alumina crystals. As a result of this flattening, the sintered alumina grains and the areas with prominent grains are indistinguishable from those of the control surfaces (Fig. 4, *B* and *D*). Awliya et al[26] also showed that airborne particle abrasion promoted a blunting of the microstructure surface of Procera. However, it should be noted that the shear bond strength between Procera ceramic and resin luting agent was higher when airborne particle abrasion was applied on the surface than the shear bond strength obtained with hydrofluoric acid etching.[26]

This study has shown that the efficiency of the surface treatment is highly dependent on the composition of the ceramics. Both hydrofluoric acid etching and airborne particle abrasion promoted irregularities in IPS Empress, IPS Empress 2, and Cergogold. These irregularities may be instrumental in improving the bond strength with resin luting agents.[13,26] The etching times recommended by the manufacturer and followed in this study were of sufficient duration to produce morphologic change of these ceramics. For the In-Ceram Alumina, In-Ceram Zirconia, and Procera, neither the hydrofluoric acid nor the airborne particle abrasion was effective in increasing irregularities on the ceramic surface. The etching protocol used in this study (2 minutes) was not of sufficient duration to produce evidence of morphological change of the ceramic surface. In this situation, a retentive preparation design coupled with an adhesive luting agent is likely to be needed to secure the ceramic restoration.

CONCLUSIONS

Within the limitations of this in vitro study, the following conclusions are drawn: Hydrofluoric acid etching and airborne particle abrasion with 50-μm aluminum oxide increased the irregularities on the surface of IPS Empress, IPS Empress 2 and Cergogold ceramics. Hydrofluoric acid and airborne particle abrasion with 50-μm aluminum oxide did not change morphologic microstructure on the surface of In-Ceram Alumina, In-Ceram Zirconia and Procera.

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