Shear bond strength of a resin cement to densely sintered high-purity alumina with various surface conditions

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Procera Sandvik AB is now manufacturing a densely sintered high-purity alumina core for an all-ceramic crown designed for anterior and posterior restorations. Whereas the material holds promise on the basis of in vitro strength tests, the ability to alter the surface and use conventional bonded resin cements has not been reported previously in the literature. Samples of the core were treated by means of one of four methods routinely used for all ceramic restorations, and then a commercially available resin cement was bonded to the surface. A shear bond test of the adhesion showed that the highest shear bond strengths of 11.99 ± 3.12 MPa were obtained with air abrasion at 80 psi and 50-μm alumina particles. □ *Ceramic; dental cements; dental materials; silane; surface treatment*

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The increased desire for optimum esthetics in restorative treatment has made the all-ceramic crown a frequently used alternative for both anterior and posterior restorations. Although several systems are available to provide an acceptable all-ceramic crown, the dental profession continues to look for the ideal, which would combine preferred esthetics with excellent strength and marginal fit properties (1, 2).

The first forms of ceramic crowns were pleasing and matched the surrounding dentition; however, their applications were limited owing to their inherent weakness. In an effort to improve the strength and increase versatility, several substrates (or core materials), such as Vita In-Ceram, Dicor, Hi-Ceram, and IPS-Empress, have been developed, to which conventional porcelains may be added.

Long-term clinical success of totally ceramic crowns seems to be dependent on successful bonding to large areas of prepared dentin. Jensen et al. (3) showed that significant improvement in fracture resistance of ceramic restorations can be achieved by using adhesive bonding techniques. Eden & Kaztez (4) showed a significant increase in resistance to fracture of bonded Dicor crowns compared with identical crowns luted with a conventional zinc phosphate cement. Although critical for the long-term success, the bonding usually requires a modification of the porcelain surface to achieve high bond strength. Modification of the ceramic surface is not always possible or requires extreme measures, making the process impractical (5–8).

A new technique for manufacturing an individual all-ceramic crown built on a coping of densely sintered high-purity alumina, Procera All Ceram, has recently been developed by Nobel Biocare AB (previously Nobelpharma AB) and AB Sandvik Hard Materials, Sweden (9). The technology shows promise, but it has not been reported whether it is possible to use bonded resin cements with this crown.

The aim of this study was to evaluate the influence of surface treatment of the densely sintered high-purity alumina as used in the Procera All Ceram coping before bonding and to evaluate the influence of such treatments on the bond strength of a resin cement to this aluminous core material.

Materials and methods

Cylindrical specimens 6 mm in diameter and 8 mm long were manufactured from high-purity densely sintered alumina by Procera Sandvik AB (Stockholm, Sweden). The flat surface used for the shear bond test was ground with a diamond wheel. The samples were divided into 4 groups of 10 each, and the flat surface used for the shear bond test was treated as follows: 1) Etching with 9.6% hydrofluoric acid for 2 min, followed by thorough rinsing for 30 sec using air/water spray. 2) Sandblasting with 50-μm alumina particles for 15 sec with a micro-etcher, followed by air-spraying. 3) Roughening with a diamond and etching with 37% phosphoric acid for 2 min, followed by thorough rinsing for 30 sec with water/air spray. 4) No treatment (control).

All samples were examined in a scanning electron microscope (SEM) for determination of surface morphology.

The second phase of the experiment examined the effect of the above four surface treatments on shear bond strength of a dual-cure resin cement to the alumina core.

To facilitate insertion of the samples in the Instron

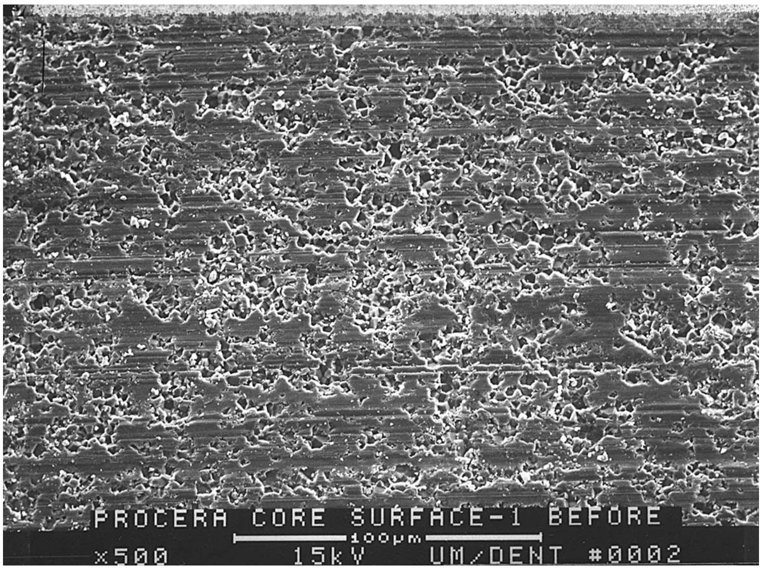

Fig. 1. Scanning electron microscope photograph of the flat surface of alumina used for the shear bond test. The ground surface appears rough, with numerous grains pulled out and traces from the diamonds in the grinding wheel.

testing machine, 40 samples were embedded in stone, using stainless steel rings 30 mm in diameter and 30 mm high. The surface of the samples was mounted flush with the surface of the stone. Samples were then divided into 4 groups of 10 each and treated as previously described. After surface treatments, the areas for bonding were isolated by means of one-sided adhesive tape with a pre-punched circular hole (4 mm in diameter). The purpose of isolating the bonded area was to prevent excess flash adhering to the surrounding core material, which could produce inflated bond strengths. The exposed sample surfaces were coated with Enforce silane coupling agent (Dentsply Caulk, Milford, Del., USA) for 1 min. Enforce Bonding agent and the Enforce dual-cure resin cement were applied to the surface of all samples by using an aluminum mold with a diameter of 4 mm at the bond interface and 4 mm thick. The resin was applied and photopolymerized in accordance with the manufacturer's directions and kept in 100% humidity at room temperature for 7 days. After storage, each sample was subjected to a shear load in a Universal testing machine (Instron Corp., Canton, Mass., USA) at a cross-head speed of 0.5 mm/min and using a knife-edge blade placed parallel to the bonded surfaces. Recordings were made of shear load at the point of failure, and shear bond strengths were calculated by dividing the force at which bond failure occurred by the bonding area. From these data the mean and the standard deviation for each group were calculated.

The data were analyzed with one-way analysis of variance (ANOVA) to determine whether significant differences existed in the shear bond strengths of the resin cement to densely sintered high-purity alumina with and without surface treatments, and whether there was a difference among the different surface treatments.

Results

Representative scanning electron microscope (SEM) examinations of the densely sintered high-purity alumina core surface before surface treatments are illustrated in Figs. 1 and 2. The microstructure of the ground surface of the alumina core appears relatively rough, with some grains pulled out as a result of the grinding with the

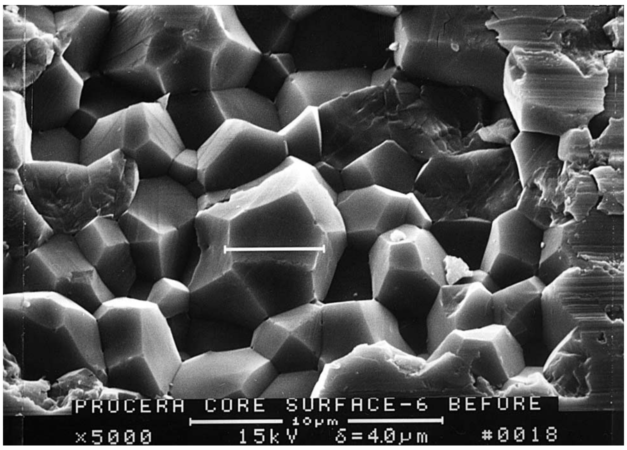

Fig. 2. A closer look at an area with grain pull-outs. The alumina grains are easily seen in this photograph.

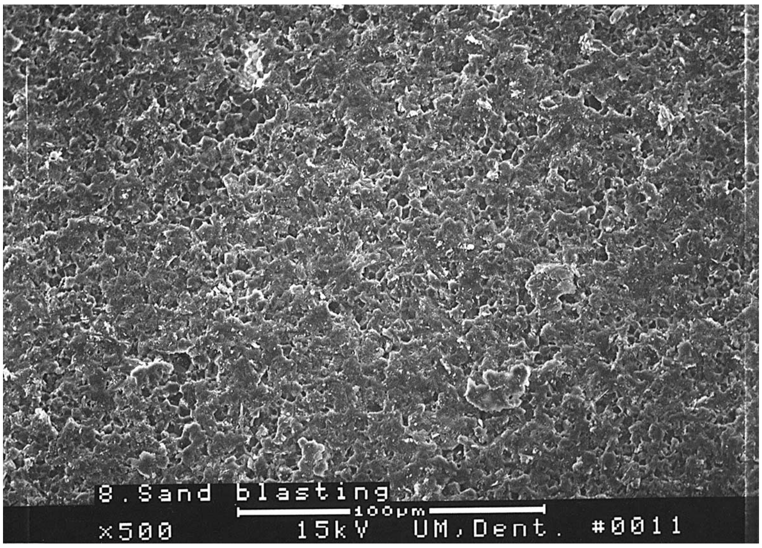

Fig. 3. Scanning electron microscope photograph of alumina oxide core material after sandblasting with 50-μm aluminum oxide.

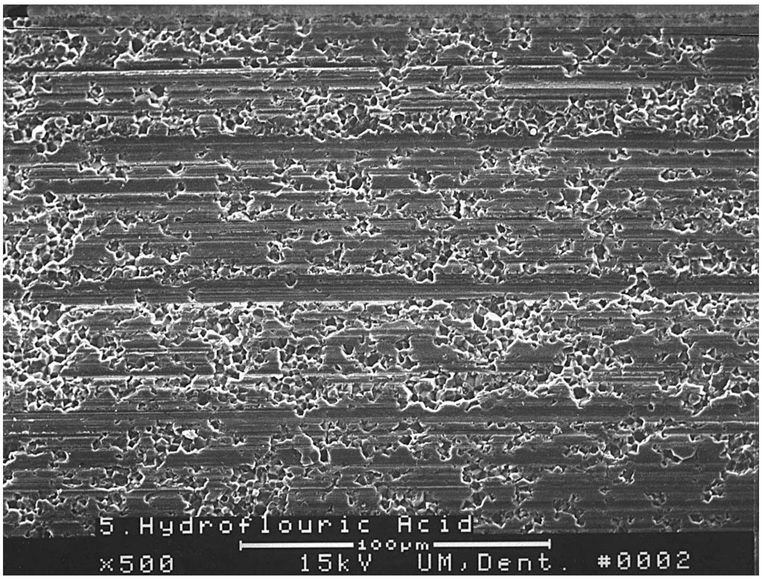

Fig. 4. Scanning electron microscope photograph of alumina after treatment with 9.6% hydrofluoric acid.

diamond wheel (Fig. 1). At higher magnification of one area grain pull-out and the shape of the grains of densely sintered alumina are easily identifiable (Fig. 2). When the surface was sandblasted with 50 μm alumina, using a micro-etcher, all sharp edges in the microstructure were blunted, and as a result the grain boundaries of the alumina and areas with grain pull-out were not as distinct as those of the ground surface (Fig. 3). Similar characteristics are not evident when the surface of the alumina core was etched with the 9.6% hydrofluoric acid (Fig. 4). The alumina grain boundaries after etching appear as distinct as before treatment, suggesting almost no surface degradation. When the surface was roughened with a diamond instrument, it appeared similar to the surface treated with the hydrofluoric acid. However, the diamond instrument caused some rounding of the alumina grain boundaries in grain pull-outs caused by grinding and in addition created new grain pull-outs (Fig. 5).

Table 1 illustrates the shear bond strengths of Enforce resin cement to the alumina core treated with the four different methods. The highest shear bond strength obtained was after sandblasting the surface with 50-μm

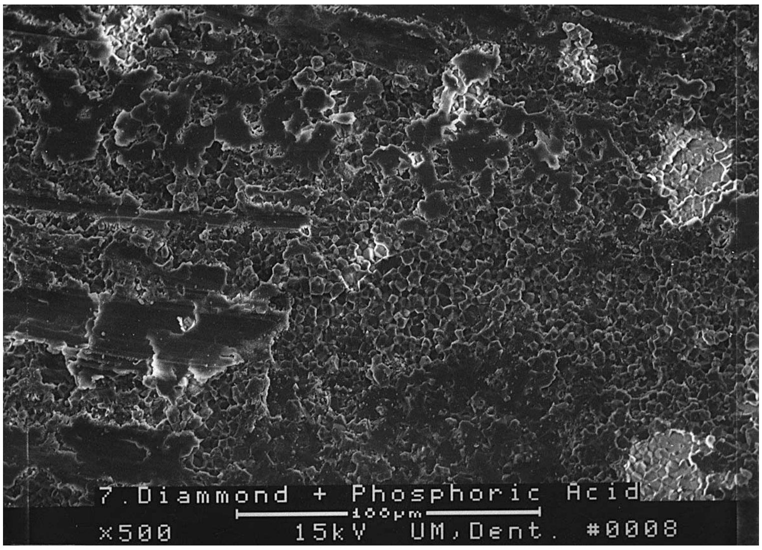

Fig. 5. Scanning electron microscope photograph of alumina after treatment with a diamond bur and 37% phosphoric acid.

alumina particles by means of a micro-etcher. The lowest shear bond strength of resin cement was to the hydrofluoric acid-etched surfaces.

One-way ANOVA shows a statistically significant difference at the 25% confidence level ($P < 0.001$) of the bond strengths among the groups with regard to the four surface treatments. Tukey's multiple comparison was computed to achieve a multiple comparison of the four different surface treatments used in the second phase of the study (no treatment, hydrofluoric acid, sandblasting with a micro-etcher, and diamond abrasion). The different comparisons indicated that sandblasting the surface of the alumina core with 50-µm alumina particles was significantly better than the other three treatments (versus no treatment and hydrofluoric acid ($P < 0.001$) and versus diamond abrasion ($P < 0.05$)). There was no significant difference between no treatment and diamond abrasion and hydrofluoric acid, respectively ($P > 0.05$), whereas diamond abrasion was better than hydrofluoric acid.

Discussion

A strong and permanent bond between hard dental tissues and restorative materials provides improved marginal adaptation, thereby preventing micro-leakage resulting in pulpal sensitivity or penetration of bacteria and toxic substances and discoloration. It has been repeatedly shown that resin bonding increases fracture resistance of the restorations and may minimize tooth preparation with less removal of dental tissues (10–13).

Table 1. Shear bond strength, mean and standard deviation (*s*) in (MPa), of resin cement (Enforce) to the four different surface treatments of densely sintered high-purity alumina

Groups	(*n*)	Mean	*s*
Hydrofluoric acid	10	5.38	1.28
Sandblasting	10	11.99	3.12
Diamond abrasion + phosphoric acid	10	9.13	1.92
Control (no treatment)	10	6.66	1.58

The SEM examination of this densely sintered high-purity alumina core material showed the grains and the junctions between the grains of the alumina particles. No pores were seen within the core material, which agrees with the findings of Coble (14, 15). He proposed that during sintering to full density, pores will follow the movement of the grain boundaries and will not become trapped inside the grains. SEM examinations of the test samples showed some roughness and grain pull-out on the surface of the specimens with no treatment. These grain pull-out areas are a result of grinding the surfaces during the manufacturing of the samples.

The present study investigated the effect of different surface treatments on shear bond strengths of resin cement to the alumina core. The data clearly showed that sandblasting the surface with alumina particles was the most effective surface treatment for producing high bond strengths. Shear bond strength results after a surface treatment with 9.6% hydrofluoric acid were substantially lower than after sandblasting. These results do not correspond with those of previous studies (16, 17), which showed maximum bond strength values with hydrofluoric acid. On the other hand, these results correspond to the results of Sorensen & Engelman (18), comparing the effect of hydrofluoric acid etching on shear bond strength of resin cement to porcelain. Various feldspathic porcelains with low alumina (10%), medium alumina (20%), and high alumina (30%) were tested before and after etching with

20% hydrofluoric acid. Their results showed that hydrofluoric acid etching significantly increased the bond strength of most feldspathic porcelains with low and medium alumina but did not improve the bond strength of the core porcelain with high alumina content.

Differences in composition and microstructure of the materials and the resulting surface morphology after etching are important factors in explaining the difference in bond strength of various ceramic materials. Densely sintered high-purity alumina contains only one phase, and the acid etching will only affect the grain boundaries visible on the surface. It has recently been proposed that improved silane agents may make it possible to eliminate acid etching from the all-ceramic cementation protocol (19).

The significant difference in bond strength of resin cement to the alumina core surfaces after sandblasting or acid etching might also be explained by the differences in the microstructure of the surface after treatment. SEM observations indicated more grains pulled out, giving undercuts of the sandblasted surfaces, than on the acid-etched surfaces. Differences in surface topography after the two treatments may be responsible for developing a higher bond strength with sandblasting than can be achieved with hydrofluoric etching.

The exact bonding mechanism of silane to the alumina core is not fully understood. Silane coupling agents or a porcelain primer was applied on the surface of the core, following the manufacturers' instructions for porcelain bonding. One of the mechanisms postulated that silane bonding is mediated through the silica at the surface of porcelain (17). However, the action of silane on the surface of the alumina core is limited because the silane bond to alumina is low. Kern & Thompson (8) found that application of silane on surfaces of sandblasted In-Ceram did not provide a stable bond, which they attributed to the small percentage of silica used for In-Ceram and to the weak and unstable bond between alumina and silane coupling agent. The silane used in this study may have acted as a wetting agent for the resin cement and by means of that mechanism promoted adhesion.

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