

ORIGINAL ARTICLE

Bond strength of a composite luting agent to alumina ceramic surfaces

TORE DERAND^{1,2}, MARGARETA MOLIN^{1,2} & KETIL KVAM²

¹Dental Technology and Dental Material Science, Faculty of Odontology, Malmö University, Sweden, and ²Scandinavian Institute of Dental Materials, Oslo, Norway

Abstract

Objective. The purpose of this study was to evaluate the shear bond strength (SBS) of a dental luting agent to alumina ceramics after different surface pretreatment. **Material and Methods.** Specimens ($n=50$) of pressed blocks ($10 \times 0 \times 5$ mm) of alumina ceramic (Procera AllCeram) were divided into untreated specimens (AF) as provided by the manufacturer and polished specimens (AP). Five groups of specimens ($n=5 \times 10$) with different surface pretreatments were prepared. Groups 1 and 2: AF and AP without any pretreatment; Group 3: AF treated with silane, (AF-s); Group 4: AF treated with RF plasma spray (AF-RF); Group 5: AF treated with low fusing porcelain (AF-p) glass pearls. Composite cylinders (5×5 mm) were cemented to the test specimens with a resin luting agent. The specimens were loaded to failure in shear mode using a universal testing machine. Recorded loads were used to calculate SBS in MPa. The results were analyzed using one-way ANOVA and the Tukey HSD multiple comparison test at $\alpha=0.05$. Scanning electron microscopic micrographs (SEM) were used to characterize surfaces of interest. **Results.** Polished surfaces had significantly lower SBS ($p < 0.05$) compared with untreated specimens (AP vs AF). Silanated, non-polished surfaces (AF-s) revealed lower SBS, even though the result was not significantly different from that of AF-s without silane treatment. Plasma treatment improved SBS by a factor of 2 ($p < 0.05$) and treatment with low-fusing porcelain micro pearls increased SBS by a factor of 3 compared to untreated surfaces ($p < 0.05$). The layer of glass pearls did not exceed $5 \mu\text{m}$ (SEM). **Conclusions.** Within the limitation of the conditions of this study, treatment of alumina oxide ceramic surfaces with a plasma spray coating or a low-fusing porcelain pearl layer significantly increased the SBS of a resin luting agent to the ceramic surface.

Key Words: Bonding, dental ceramics, resin, shear test

Introduction

The use of all-ceramic restorations is rapidly increasing in clinical treatment. The development of high-strength ceramics has extended the use of all-ceramic crown restorations and short-span posterior fixed partial dentures [1]. Catastrophic failures have decreased with the introduction of core materials made of densely sintered alumina [2]. Historically, most high-strength ceramic restorations have been cemented to a tooth with zinc phosphate or glass-ionomer cements, but now resin luting agents are the first choice as they offer advantages such as good retention, low solubility, low film thickness, and good marginal adaptation [3]. A great number of luting cements and various surface treatments have been proposed to improve bond strengths to ceramics [4]. However, there is still some clinical concern about obtaining a reliable and strong bond

between a composite luting agent and high-strength ceramics such as alumina and zirconia.

Aging and thermal cycling may decrease the bond strength of resin luting agents to ceramics over time [5,6]. The retention of resin luting agents on unaltered ceramic materials depends primarily on micro-mechanical surface irregularities [7]. Etching with hydrofluoric acid has therefore been successful on surfaces of ceramic materials containing a glass component, but the acid has no influence on pure alumina ceramics and so for these materials, the retentive quality is unaltered [8]. One method for improving retention for alumina ceramics is to roughen the surface with airborne-particle abrasion [9]. Good retention requires resin bonding to both ceramic and dentin/enamel [10].

The use of silanes is recommended for glasses containing silicon with which a chemical reaction may occur and a siloxane (-Si-O-Si-O-) network

with covalent bonding and perhaps an element of hydrogen bonding is established with the glass surface. That is not the case with ceramics with high alumina content, although one report showed doubling in bond strength after silanization, but no explanation was given as to why this occurred [11]. The application of silane has been controversial with regard to the effect of the adherence between composite materials and these types of ceramic surfaces [12]. However it has also been suggested that silanization may improve the wetting of the surface, resulting in higher bond strength values even though very small [13].

The plasma spray technique is used in several areas, and for medical and surgical equipment it is used to coat alloys, metal, and ceramics. Plasma is a partially ionized gas containing ions, electrons, atoms, and neutral species. To enable the gas to be ionized in a controlled and qualitative manner, the process is carried out under vacuum conditions. A high-frequency generator which can be in the kHz, MHz, or microwave range, is then used to ionize the gas into plasma. Plasma-spray techniques can give a good bond even if the bonding energy is still difficult to explain. This technique has been tested together with various materials in various situations [14–16] and the superior bond provided to several materials can probably be explained by established covalent bondings [17].

Improved mechanical interlocking of resin cements could be attained by fusing micro pearls of porcelain on the intaglio surface of glass or ceramic materials. These may attach to the aluminium oxide and create a mechanical base for retention of the resin luting cement. However this technique must not interfere with the conventional firing scheme of dentin and enamel porcelain and the thickness must be less than 10 µm in order that it does not disturb the fit of the restoration [18,19].

The aim of this study was to investigate whether pretreatment with plasma spray and fused glass pearls to alumina ceramic surfaces improves the shear bond strength (SBS) of the dental resin agent. The hypothesis was that these treatments should increase the bonding.

Material and methods

The test material consisted of pressed blocks (10 × 10 × 5 mm) of alumina (Procera AllCeram; Nobel Biocare, Sweden). Two types of blocks were used: untreated blocks as delivered from the manufacturer (AF), and blocks polished with sandpaper to an even and shiny surface (AP) (Figure 1). The surfaces were cleaned with ethanol and air dried before surface treatment.

Five groups of specimens ($n=5 \times 10$) were prepared to accept variable surface treatments. Pressed and polished specimens were used in two groups

(AF, AP). In one group silane treatment (Ultradent; Ultradent Products, South Jordan, USA) was used for 15 s and dried for 20 s (AF-s). In another group, radio frequency (RF) plasma treatment (hexamethyldisiloxane (HDMSO) at 13.56 MHz) was performed, using a reactor from Plasma Electronic (Neuenburg, Germany) (AF-RF). The plasma deposition was made in three sequential steps: 1) activation of the surfaces with oxygen; 2) deposition of HDMSO ((CH₃)₃SiOSi(CH₃)₃); and 3) activation of the polymer with oxygen.

In the last group, the blocks were treated with low-fusing enamel porcelain powder (Duceratin; Ducera, Rosbach, Germany) (AF-p). The powder was stirred in an excess of water and immediately painted on the ceramic surfaces and fired at 720°C without a vacuum.

Test cylinders were constructed of polymer tubes made of polymethylmethacrylate with an inner diameter of 5 mm and 5 mm in height. The tubes were filled with composite (Charisma; Heraeus Kulzer, Dormagen, Germany) and cured for 40 s. Adhesive tape (Scotch Tape, 3 M, Minn., USA) with a 5 mm-diameter circular hole was placed on the ceramic surface and thereafter the test cylinder was cemented into the hole. This was done to facilitate removal of excess resin agent around the cylinder after hardening. The composite cylinder was cemented by applying a drop of resin agent (Variolink II; Ivoclar-Vivadent, Schaan, Liechtenstein) on the ceramic surfaces, after which the cylinder was pressed on the ceramic block at a load of 0.3 kg. The resin agent was light-polymerized from three directions for 40 s each. The specimens were kept in air for 1 h before testing.

The SBS until failure was determined using a universal testing machine (LRX; Lloyd Instruments, Fareham, England) with a crosshead speed of 0.5 mm/min (Figure 2). Selected ceramic surfaces and the fracture surfaces of the test cylinders were analyzed using scanning electron microscopy (SEM). The thickness of the layer of glass pearls was measured using both a precision micrometer and SEM micrographs.

The mean values of each group were statistically analyzed using one-way analysis of variance (AN-OVA) and the Tukey HSD multiple comparison test at $p < 0.05$.

Results

The results of the shear bond test are presented in Table I. The AP provided significantly ($p < 0.05$) lower SBS values compared with the AF. Silane (AF-s) yielded lower but not significantly lower mean values compared with AF. The SBS values of plasma-sprayed unpolished surfaces (AF-RF) were twice ($p < 0.05$) the values found for AF. SBS values

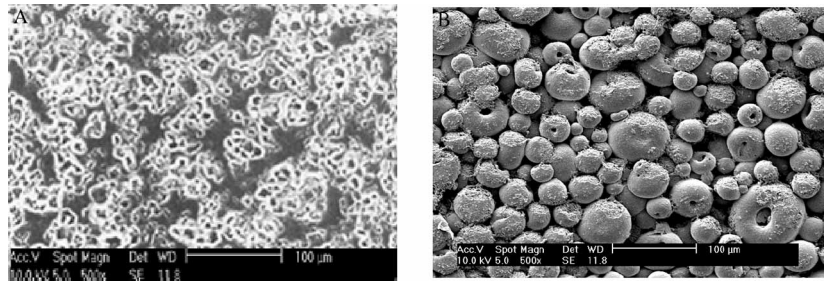


Figure 1. A. Flat polished alumina ceramic surface. B. Alumina ceramic unpolished surface.

were tripled ($p < 0.05$) for surfaces treated with glass pearls (AF-p) compared with AF.

SEM revealed no signs of resin on the ceramic surfaces after debonding and the glass pearls were still attached to the ceramic surfaces. Glass pearls fused onto the surface of the alumina block and corresponding composite test cylinder are shown in Figure 3, focusing on the glass pearls. The thickness of the layer of pearls did not exceed 5 μm .

Discussion

In the present study shear bond tests were used to evaluate the bond strength of a resin agent to alumina ceramic after two different surface treatments. The test method is fast and easy to perform but it can be questioned whether a tension test might be more appropriate for testing the adhesive capabilities of resin agents to ceramics [20]. However, as the intention was to evaluate relative differences in pretreatment techniques and how they could result in improved bonding, which may be of clinical interest, the method used was thought to be relevant. It has been shown in several studies that thermocycling and long-time storage in water decrease bond strength between resin luting materials and ceramic surfaces as well as tooth materials [5,6]. In the present study the main purpose was to examine the

principal improvements in bond strength after surface treatment. Thus, as a first step, only artificial materials and no aging processes were used to study the bonding strength. Furthermore, ceramic constructions are subjected to high loads immediately after placement, so it is important to study the bond strength after surface pretreatment and without simulated aging processes. With the fabrication of the alumina cores, some surface irregularities arise and it was expected that the retention of the resin agent should be better than when even and polished surfaces were tested. This difference was also found to be statistically significant.

The tested ceramic did not contain any silica, so no bonding to silane could be expected, and in this study no significant effect of silanization was found at pressed surfaces but a tendency towards reduced bond strength values could be detected. The plasma treatment showed a doubled increase in bond strength but what happens in the bonded area is not known but it is possible that more than one type of electronic and covalent bonding may occur. In the SEM micrographs no sign of the HDMSO layer could be detected and it was concluded that the thickness must be below 0.5 μm .

The technique of fusing glass pearls to the ceramic surface created a mechanical interlocking of the resin agent layer. The shear bond test showed that the bond strength was tripled and the fracture between the resin cement and the ceramic was adhesive. The superior retention was obviously connected to the macro-mechanical retention caused by the glass pearls compared to the micro-mechanical retention

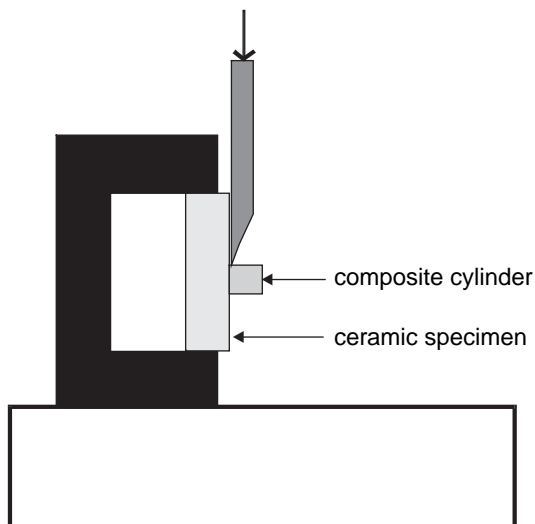


Figure 2. Test apparatus for shear bond tests.

Table I. Significant differences ($p < 0.05$) in shear bond strength (MPa) of composite specimens on aluminium oxide surfaces

Test group	<i>n</i>	M	SD	Significance level
AF	10	7.1	1.3	NS
AF-s	10	5.3	0.7	
AP	10	2.1	0.3	
AF-RF	10	15.2	2.6	
AF-p	10	21.1	3.5	

M = mean; SD = standard deviation; *n* = number of specimens; NS = no significance ($p > 0.05$); AF = pressed alumina; AP = polished alumina; AF-s = pressed alumina painted with silane; AF-RF = pressed alumina plasma treated; AF-p = pressed alumina with glass pearls.

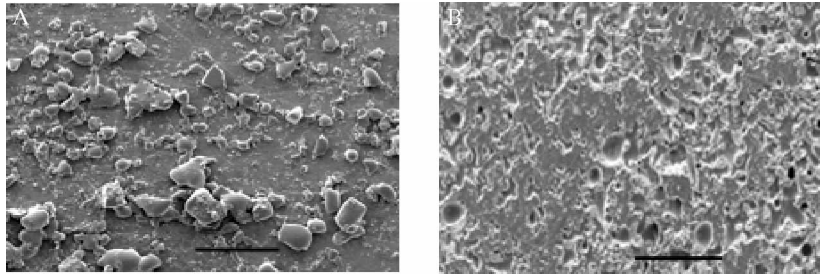


Figure 3. A. Surface with fused glass pearls. B. Corresponding surface in composite, line = 5 μm .

in untreated alumina surfaces. The technique of fusing glass pearls to the surface could be done as a final treatment or probably combined with the glazing of the crown or bridgework. In this experiment, low-fusing porcelain powder was used and the handling resulted in separate glass pearls spread out over the ceramic surface so that the layer did not exceed 5 μm in thickness. It is important that the pearls do not interfere with the fitness of the core but the thickness of resin agent layers are mostly between 20 and 50 μm [19]. The pearls were so firmly attached to the alumina ceramic that the fracture was always seen at the resin layer and no pearls could be detected in the resin surface. Further studies are ongoing to optimize bond capacity and cements with good wetting of the pearls attached to this type of core material. Further improvements in the combination of glass pearls and silanization may increase the attachment and result in an even higher bonding strength of the resin agent.

Both methods examined here are quick to perform and require only short handling procedures.

Conclusion

Within the limitation of the experimental conditions of this study, treatment of alumina oxide ceramic surfaces with plasma-spray coating or a low-fusing porcelain pearl layer significantly increased the SBS of a resin luting agent to the ceramic surface.

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