

The effect of alumina and aluminium nitride coating by reactive magnetron sputtering on the resin bond strength to zirconia core

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PURPOSE. Although several surface treatments have been recently investigated both under *in vitro* and *in vivo* conditions, controversy still exists regarding the selection of the most appropriate zirconia surface pre-treatment. The purpose of this study was to evaluate the effect of alumina (Al) and aluminium nitride (AlN) coating on the shear bond strength of adhesive resin cement to zirconia core. **MATERIALS AND METHODS.** Fifty zirconia core discs were divided into 5 groups; air particle abrasion with 50 µm aluminum oxide particles (Al₂O₃), polishing + Al coating, polishing + AlN coating, air particle abrasion with 50 µm Al₂O₃ + Al coating and air particle abrasion with 50 µm Al₂O₃ + AlN coating. Composite resin discs were cemented to each of specimens. Shear bond strength (MPa) was measured using a universal testing machine. The effects of the surface preparations on each specimen were examined with scanning electron microscope (SEM). Data were statistically analyzed by one-way ANOVA (α=.05). **RESULTS.** The highest bond strengths were obtained by air abrasion with 50 µm Al₂O₃, the lowest bond strengths were obtained in polishing + Al coating group (*P*<.05). **CONCLUSION.** Al and AlN coatings using the reactive magnetron sputtering technique were found to be ineffective to increase the bond strength of adhesive resin cement to zirconia core. [J Adv Prosthodont 2013;5:382-7]

KEY WORDS: Air abrasion; Magnetron sputtering; Coating; Zirconia

INTRODUCTION

Developments in the ceramic materials science for dental applications has increased the popularity of high strength core-veneered all-ceramic restorations (alumina and zirconia based ceramics) in the last few years. One of the high

strength material is yttria stabilized tetragonal zirconia polycrystals (Y-TZP, zirconia).¹

High flexural strength and fracture toughness, white color, chemical and structural stability and combined with CAD/CAM technology afford its application as framework material for fixed partial dentures (FPDs) without any limitation regarding the size even in loaded reconstructions in the posterior region.² Due to their high fracture resistance, zirconia crowns and FPDs can be cemented using conventional luting methods recommended by the manufacturers.³ However, adhesive luting techniques can provide significant clinical advantages over conventional cementation of dental restorations. Adhesive luting is advocated for improving the retention, marginal adaptation, fracture resistance of restorations and inhibition of secondary caries.^{1,3,4} Although superior in terms of mechanical performance (strength, toughness, fatigue resistance), bonding of resins to zirconia core materials is more difficult than it is for conventional

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silica-based dental ceramics.⁵⁻⁷

Chemo-mechanical and mechanical surface treatments affect surface topography and roughness of zirconia core material. Some of these surface treatments were grinding with diamond burs, air abrasion with aluminum oxide (Al_2O_3), tribochemical silica-coating (CoJet, Rocatec), acid etching with hydrofluoric acid, coupling with silane, plasma spraying with hexamethyldisiloxane, internal coating with low fusing porcelain pearl layer, selective infiltration etching and combinations of any of these methods.^{1,3,4,8-15} Although etching the inner surfaces of conventional Silica based dental ceramics with hydrofluoric acid or the application of most often used silane coupling agent, 3-methacryloxypropyltrimethoxysilane (3-MPS), was an effective conditioning method for bonding resin composite,^{1,16-18} these methods showed unsatisfactory resin bond to oxide based dental ceramics.^{1,6,16,19,20}

Airborne particle abrasion with 50-110 μm alumina particles at 0.25 MPa has been found to be effective in cleaning and roughening the surface of various dental oxide ceramics.^{1,13,21-23} However, currently there is a controversy whether airborne particle abrasion alters the strength of oxide ceramic restorations on a clinically relevant level.¹ Some studies showed even a strengthening effect of airborne particle abrasion on oxide ceramics,^{24,25} others reported a strength reducing effect.^{26,27} However, particle abrasion and adjusting with burs of zirconia results in creation of sharp cracks and structural defects that render the zirconia framework susceptible to radial cracking during function.^{3,15}

Some studies have shown that tribochemical silica coating increased the bond strength to oxide ceramics.^{13,22} However it has also been reported that tribochemical silica coating might be less effective for densely sintered ceramics than for glass-infiltrated ceramics.²⁸ Although several surface treatments have been recently investigated both under *in vitro* and *in vivo* conditions, concerns still exist regarding the selection of the most appropriate zirconia surface pre-treatment.

Magnetron sputtering is one of the most commonly used methods for the deposition of thin film materials. Its popularity is derived from the simplicity of the process, versatility of the technique, and flexibility with regard to system alteration and configuration.²⁹ Sputtering techniques have shown that with various thin-film materials, a uniform coat and a dense crystal surface structure with the substrate material can be achieved, even at a low temperature.³⁰ Coating by the sputtering technique has often been applied to dental alloys in order to improve the properties of the metallic dental alloy, giving the alloy a high degree of hardness, high wear and corrosion resistance and good biocompatibility.³⁰⁻³²

Recently, coating of dental ceramic surface was developed to improve mechanical properties and to obtain strong and durable resin bond strength to ceramic. The use of the magnetron sputtering technique to deposit yttria-stabilized zirconia (YSZ) thin films on dental porcelain shows promising results. This technique could be applied to all

ceramic materials in principal, to modify inherent surface flaws, suppress crack propagation, thereby improving fatigue behavior and fracture resistance, and potentially having a broader application than developing new materials and material systems.³³

The purpose of this study was to evaluate the effect of alumina (Al) and aluminium nitride (AlN) coating by reactive magnetron sputtering technique on shear bond strength of a composite resin luting cement to a zirconia core. The research hypothesis tested was that alumina and aluminium nitride coating would improve the shear bond strength of resin cement to zirconia core.

MATERIALS AND METHODS

Fifty zirconia core specimens (10 mm diameter and 2 mm height) were produced by a copy-milling system (Zirconzahn, Bruneck, Italy) using prefabricated blanks of zirconia (ICE Zircon Translucent; Zirconzahn, Bruneck, Italy) and then sintered according to manufacturer instructions.

Subsequently, all specimens were divided into 5 groups, each containing 10 specimens for surface treatments. The groups include; air particle abrasion with 50 μm Al_2O_3 (Group A), polishing + Al coating (Group PAI), polishing + AlN coating (Group PAIN), air particle abrasion with 50 μm Al_2O_3 + Al coating (Group AAI) and air particle abrasion with 50 μm Al_2O_3 + AlN coating (Group AAIN). Polishing procedure was started with a 600-grit silicon carbide abrasive paper (3M ESPE, St. Paul, USA) and finished with 1200 grit each for 10 seconds on a 300 rpm grinding machine (Buehler Metaserv, Buehler, Germany) under running water. Air particle abrasion procedure was performed using an intraoral air abrasion device (Microetcher, Danville Engineering Inc., San Ramon, CA, USA) at an air pressure of 2.5 bars for 10 seconds at a distance of approximately 10 mm.

Al and AlN coatings were deposited by a dual magnetron sputtering system and deposition conditions are summarized in Table 1. Surface morphology of zirconia specimens after coating procedure and thickness of the coatings deposited on the specimens were evaluated by scanning

Table 1. Deposition parameters

Coating	Al	AlN
Bias voltages (-V)	80	70
N2 pressures (mtorr)	-	0.5
Total working gas pressures (Ar) (mtorr)	2.0	-
Total working gas pressures (Ar + N2) (mtorr)	-	2.0
Target power (W)	1500	2000
Coating time (min)	60	60
The thickness of the coating ($\mu\text{m}/\text{nm}$)	1.5	400

electron microscope (SEM-Leo 440, Cambridge, England).

Fifty composite resin discs (Filtek Z250, 3M ESPE, Seefeld, Germany) were fabricated by compact the material into a polytetrafluoroethylene mold (Isoflon, Diemoz, France) with a hole in center (6 mm diameter and 2 mm thickness). Composite resin was incrementally condensed into the mold to fill up the mold and each layer was light polymerized for 40 seconds at a distance of 1 mm using a light- polymerizing unit (Astralis 3, Ivoclar Vivadent, Schaan, Liechtenstein) with an output power of 600 mW/cm². One composite resin block was fabricated for each specimen.

Composite resin discs were cemented to the specimen surfaces with a dualpolymerized adhesive resin cement (Panavia F 2.0, Kuraray, Osaka, Japan). For cementation, equal amounts of a dual-polymerized resin luting agent paste base and catalyst were mixed and applied to the composite resin block with a plastic spatula. Each composite disc was bonded to a zirconia core specimen under the load of 500 g. The excess resin cement was removed by means of a brush. The resin cement was then light polymerized for 20 seconds with a curing light (Astralis 3). A glycerin gel (Oxyguard II, Kuraray, Osaka, Japan) was applied for 10 minutes.

Specimens were embedded in the centers of autopolymerizing acrylic resin blocks (Meliodent, Heraeus Kulzer, Armonk, NY, USA) then were stored in distilled water at 37°C for 24 hours and thermocycled for 6000 cycles between 5 ± 2°C and 55 ± 2°C with a dwell time of 30 seconds.

A universal test machine (Lloyd LRX, Lloyd Instruments PIC., Fareham, Hampshire, England) was used for shear bond strength test at a crosshead speed 0.5 mm/min. The shear bond strength values were calculated in megapascal (MPa) by dividing the failure load (N) to the area of the

composite resin discs ($N/\pi r^2$). Data were statistically analyzed.

The Kolmogorov-Smirnov test showed that the data was of a normal distribution ($P>.05$). A homogeneity of variance test was done using Levene's test ($F: 0.301, P>.05$). Means and standard deviations of bond strengths were calculated and mean values were compared by one-way analysis of variance (ANOVA) (SPSS 12.0; SPSS Inc., Chicago, IL, USA), followed by a multiple comparisons' test performed using a Post Hoc Tukey test ($\alpha=.05$).

To assess the mode of failure, the specimen surfaces were examined with a stereomicroscope (Stemi 2000-C; Carl Zeiss, Gottingen, Germany) at a magnification of ×10. The failure modes were classified into one of the following: adhesive failure at the coated zirconia core surface; or cohesive failure in the resin cement.

RESULTS

The mean values and standard deviations of shear bond strength values of all tested groups are presented in Table 2. One-way ANOVA analysis revealed significant differences between the groups (Table 3). Air abrasion with 50 μm Al₂O₃ particles showed higher bond strength of the resin cement to zirconia core and significant differences were found when compared with Al and AlN coated groups.

Coating of zirconia core surfaces after air particle abrasion with 50 μm Al₂O₃ particles or polishing did not affect the shear bond strength of resin cement. The lowest shear bond strength value was obtained in group PAI and no significant difference was found between group PAIN.

SEM photomicrographs of air abraded or Al and AlN coated zirconia surfaces are presented in Fig. 1. Air abra-

Table 2. Mean (MPa) and standard deviation (SD) values of shear bond strength values

Groups	Surface treatment method	MPa (± SD)
Group A	Air abrasion with 50 μm Al ₂ O ₃	16.08 (± 0.75) ^c
Group PAI	Polishing + Al coating	9.18 (± 0.78) ^a
Group PAIN	Polishing + AlN coating	9.41 (± 0.61) ^a
Group AAI	Air particle abrasion with 50 μm Al ₂ O ₃ + Al coating	11.87 (± 0.80) ^b
Group AAIN	Air particle abrasion with 50 μm Al ₂ O ₃ + AlN coating	11.34 (± 0.90) ^b

* Values having same letters were not significantly different for Tukey test ($P>.05$)

Table 3. Result of one-way ANOVA

	Sum of squares	df	Mean square	F	Sig.
Between Groups	308.85	4	77.21	128.90	0.001
Within Groups	26.95	45	.59		
Total	335.80	49			

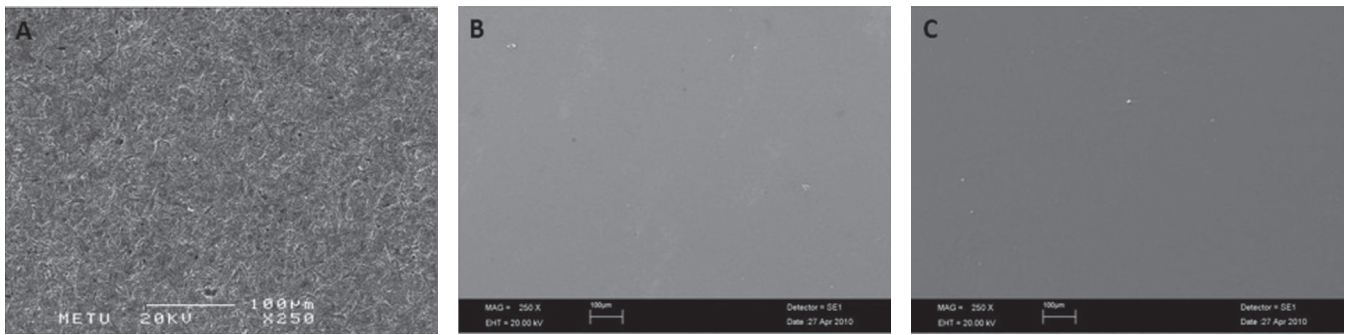


Fig. 1. SEM images of treated zirconia surfaces; Air particle abrasion with Al_2O_3 (A), Al coating (B), AlN coating (C).

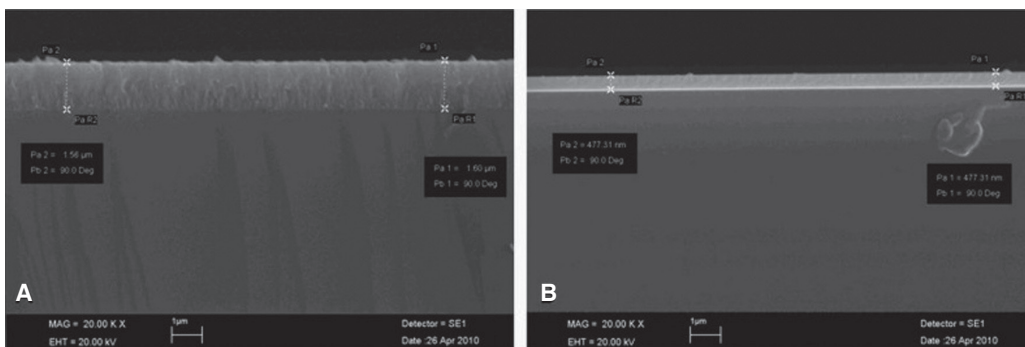


Fig. 2. Lateral view of coated zirconia surfaces; Al coating (A), AlN coating (B).

sion with $50\ \mu\text{m}\ \text{Al}_2\text{O}_3$ (Fig. 1A) created more irregular and rough surface when compared with Al (Fig. 1B) or AlN coated (Fig. 1C) specimens. Al and AlN coated specimens represented similar smooth surfaces. Fig. 2A represents lateral view of Al coating whereas Fig. 2B represents AlN coating. SEM analysis showed the coating thickness to be $1.5\ \mu\text{m}$ for Al and $0.4\ \mu\text{m}$ for AlN.

For group A with high shear bond strengths, failure mode was adhesive (60%) and cohesive in the resin cement (40%), leaving a small amount of residual resin cement on the debonded surface. The failure mode in Group PAI, PAIN, AAI and AAIN was adhesive at the coated zirconia core surface (100%).

DISCUSSION

The data support rejection of the hypothesis that Al and AlN coating would improve the shear bond strength of resin cement to zirconia core. Al and AlN coating with air abrasion or polishing, showed lower bond strength values than air abrasion with $50\ \mu\text{m}\ \text{Al}_2\text{O}_3$ particles. SEM images (Fig. 1B, 1C, 2A and 2B) revealed dense, uniform and smooth coating structure on the zirconia core surface. The result of the present study showed that Al and AlN coating with reactive magnetron sputtering technique did not provide a bonding surface yielding strong bond strength of res-

in cement.

In previous studies, various surface coatings including tin oxide, alumina (Al), gold (Au), calcium phosphates, glass composites, bioactive phosphosilicate glasses, glass-ceramics and hydroxyapatite have been reported on ceramic surfaces.^{23,29,34,35} They were also designed for mechanical reason to increase mechanical strength of ceramic used for fixed partial denture and for biological reasons, mainly to promote cell ingrowths and osseointegration of implanted ceramics.³⁵

In earlier studies, aluminous porcelain was coated with tin oxide by a reactive ion plating technique and tensile bond strength of phosphate methacrylate based dental cement to coated and uncoated aluminous porcelain was evaluated.³⁶ The result of the study indicated that reactive ion plating technique was effective technique to increase bond strength of dental cements to aluminous porcelain. In another study, aluminous porcelain was coated with tin oxide by magnetron sputtering technique and resin cement bond strength was evaluated. Similar to the previous study, coating procedure improved the bond strength.³⁷

Ruddell *et al.*²⁹ reported that mechanical properties of machinable feldspathic porcelain can be improved by the deposition of a thin film of Au, Al or AlN using radio frequency magnetron sputtering technique. The authors stated that magnetron sputtering has the ability to produce coher-

ent thin films on a dental ceramic. Although coating of ceramic surface improved the mechanical properties, it did not interfere with the optical properties of the all ceramic restoration.²⁹ In the present study Al and AlN coating was chosen, in part, because of its translucency to visible light.

When leucite reinforced feldspathic porcelain was modified through the deposition of a sputtered YSZ thin film, the flexural strength of porcelain increased.^{33,38} It is presumed that the strengthening mechanism is due to modification of surface flaws and/or surface residual stress by the applied thin film.

Teixeira *et al.*³⁸ evaluated the different YSZ thin film thicknesses (1, 3, 5 and 7 μm) on the flexural strength of a dental ceramic. The authors reported that a slight increase in strength is shown for specimens coated with a 1 μm thick thin film, but this was not statistically different than the uncoated control group. The highest mean strength value was observed for the group coated with 3 μm YSZ thin film. An increase in the thin film thickness did not result in an increase in the flexural strength of the substrate.

The clinical fit of zirconia restorations depends on the manufacturing process. Marginal and internal gaps have been reported ranging from 29 to 119 μm .^{34,39} In the present study, the coating thickness was only 1.5 μm in Al coating, 0.4 μm in AlN coating which are too lower to cause a problem in marginal and internal adaptation.

Previous studies showed that nano-structured alumina coating seems to be effective in creating microretention, providing favorable resin bonding.^{23,34} Jevnikar *et al.*³⁴ evaluated the effect of nano-structured alumina coating on resin bond strength to zirconia ceramics before and after thermocycling. The authors reported that alumina coating to Y-TZP ceramics created a highly retentive surface for resin penetration. The resin bond strength to coated groups was significantly higher than non-coated groups. Despite of morphological differences, the bond strength did not differ between coated groups (as-sintered, polished and air-borne particle abraded). In a recent study, Zhang *et al.*²³ evaluated the influence of contamination and subsequent cleaning on the bond strength and durability of adhesive resin cement to nano-structured alumina-coated zirconia ceramic. They stated that nano-structured alumina coating improves resin bonding to zirconia ceramic and eliminates the need for air abrasion before bonding.²³

Coating of materials often involve thin film processes like physical vapor deposition (PVD), chemical vapor deposition (CVD), thermal spray coating, dip/spin coating, electroplating, sol-gel deposition or laser-based processes. These processes lead to coatings with very good mechanical and physical properties.⁴⁰ In the present study zirconia core surfaces were coated by reactive magnetron sputtering technique. This technique is one of the PVD process and is often used for coating of dental materials. It has been shown that reactive magnetron sputtering has the ability to produce coherent thin films on a dental ceramic at low substrate temperatures. Furthermore these films have a significant effect on the flexural strength and flexural modulus of

a dental ceramic, depending on the coating.⁴¹

In contrast to previous studies,^{23,34} in the present study, lower shear bond strength values were obtained in coated groups when compared to air abraded group. SEM images revealed a dense, uniform and smooth coating surface on zirconia core surface after reactive magnetron sputtering. Failure modes of the PAI, PAIN, AAI and AAIN groups were predominantly adhesive and this is in agreement with shear bond strength results where low bond strength values were found for coated surface. Zhang *et al.*²³ and Jevnikar *et al.*³⁴ used suspension plasma spraying (SPS) technique to coat the specimen surfaces. SPS is a new promising processing method which employs suspensions of sub-micrometer particles as feedstock. Therefore, much finer grain and pore sizes as well as dense and also thin ceramic coatings can be achieved. The researchers obtained microporous and nano-structured lamellar surface on the zirconia specimen. In this way a large micro-retentive area was created on the specimen surfaces, with the potential for promoting resin bonding.^{23,34} In the present study flexural strength of zirconia core after Al or AlN coating was not investigated. Also different coating parameters, materials and thickness can be considered in further studies. *In vitro* studies that replicate clinical conditions are fundamental, especially for the development of new techniques, and should be performed prior to clinical studies.

CONCLUSION

Al and AlN coatings using the reactive magnetron sputtering technique were found to be ineffective to increase the bond strength of adhesive resin cement to zirconia core.

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