Retention of Zirconia Ceramic Copings Bonded to Titanium Abutments

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Purpose: *The aim of this study was to evaluate the effect of 2 surface conditioning methods and 2 luting-gap sizes on the retention and durability of zirconia ceramic copings bonded to titanium abutments.* ***Materials and Methods:*** *Zirconia ceramic copings (Camlog Biotechnologies, Winsheim, Germany) with a luting-gap size of either 30 µm or 60 µm were bonded to titanium abutments (Camlog Biotechnologies) using the composite resin cement Panavia F (Kuraray, Osaka, Japan). The bonding surfaces of the zirconia ceramic copings were either* (a) *pretreated with airborne particle abrasion and cleaned with alcohol or* (b) *just cleaned with alcohol, whereas the bonding surfaces of all titanium abutments had been abraded and cleaned. After the specimens had been stressed for either 1, 30, 60, or 150 days by water and thermal cycling, retention was measured.* ***Results:*** *The surface conditioning method, luting-gap size, and storage time significantly (*P = *.001; 3-way analysis of variance [ANOVA]) influenced retention. Air abrasion increased the retention significantly. Failure modes were predominantly adhesive. Air-abraded copings bonded with 30-µm luting gap achieved significantly greater retention than those bonded with a 60-µm luting gap.* ***Conclusion:*** *Surface conditioning methods and the size of the luting gap have a significant influence on the retention of Camlog zirconia ceramic copings bonded to Camlog titanium abutments.* INT J ORAL MAXILLOFAC IMPLANTS 2007;22:921–927

Key words: *abutment, air abrasion, resin bond, retention, titanium, zirconia*

Two goals of restorations in modern dentistry are optimal function and esthetics.[1,2] Pure titanium has been established as the material of choice for long-term osseointegration of dental implants.[3,4] Implant-supported prostheses using titanium abutments sometimes show an unnatural gray color through the soft tissue at the cervical neck.[5,6] Especially in the anterior maxilla of patients with a high smile line this is an esthetic disadvantage. New high-strength ceramic materials could be ideal for the replacement of tooth structures in terms of both function and esthetics because of their optical qualities and their significantly improved physical properties compared to feldspathic or glass ceramics.[7]

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A milestone in esthetics was the introduction of the first all-ceramic implant abutment, Ceradapt (Nobel Biocare, Göteborg, Sweden), in 1991.[5,6,8] This alumina ceramic abutment had a densely sintered, highly purified 99.5% aluminum oxide ceramic core. It was designed to fit directly onto the restorative platform of an externally hexed implant. Wohlwena et al introduced the first zirconia ceramic abutment (Zirabut; Wohlwena Innovative, Zurich, Switzerland) in 1997. Other companies followed, and today various companies have introduced industrially manufactured all-ceramic abutments made of either alumina or zirconia for their implant systems (Table 1). Furthermore, ceramic abutments can be produced by computer-aided design/computer-aided manufacturing (CAD/CAM) systems such as the Procera system (Nobel Biocare) or the DCS system (DCS Dental, Allschwil, Switzerland). For dental applications, currently the zirconia market increases more than 12% per year.[9]

All-ceramic abutments cannot be machined to the same degree of precision as metal abutments. An imprecise fit between implants and abutments often leads to abutment screw loosening and/or other clinical problems.[7] Therefore, the Camlog Zirconia Abutment (Camlog Biotechnologies, Wimsheim, Germany) consists of a titanium abutment and a zirconia coping. The 2 parts are to be bonded with a compos-

Table 1 Industrially Manufactured All-Ceramic Abutments

Brand name	Material	Manufacturer	Year introduced to the market
Ceradapt	Alumina ceramic	Nobel Biocare Göteborg, Sweden	1991
Zirabut	Zirconia ceramic	Wohlwena Innovative Zurich, Switzerland	1997
Cera Base	Alumina ceramic, titanium abutment (Bonding in the dental laboratory)	Friadent Mannheim, Germany	2000
Cercon Balance Post	Zirconia ceramic	Degudent/Ankylos Hanau, Germany	2002
ZiReal Post	Zirconia ceramic sintered to a titanium insert	3i/Biomet Palm Beach Gardens, FL	2003
Esthetic Zirconia Abutment	Zirconia ceramic	Nobel Biocare	2003
Astra Ceramic Abutment	Zirconia ceramic	Astra Tech Stockholm, Sweden	2003
Camlog Zirconia Abutment	Zirconia ceramic, titanium abutment (Fixation in the dental laboratory)	Camlog Biotechnologies Wimsheim, Germany	2003

ite resin cement (eg, Panavia F; Kuraray, Osaka, Japan) in the dental laboratory. The abutment screw seat is completely within the titanium part, compressing the metal-to-metal interface. The stability of the metal-to-metal connection between abutments and implants achieved with a defined screwing torque is well documented; wear and corrosion have not been a clinical problem.[7,10,11] If the ceramic is directly involved in the implant-abutment connection, the titanium of the implant can abrade and wear if micromotion occurs.[7,12] In addition, a metal-to-ceramic implant-abutment connection has a less accurate fit, which increases the potential risk for postinsertion problems in comparison to metal-to-metal connections.[7]

Various bonding methods to zirconia ceramic and to titanium have been reported. The use of phosphate-monomer modified composite resins (Panavia resin group; Kuraray) in combination with airborne particle abrasion of zirconia ceramic resulted in bond strengths of up to 50 MPa with no decrease in bond strength after long-term water storage and thermal cycling.[13,14] Using a phosphate-monomer modified composite resin cement (Panavia 21) in combination with airborne particle abrasion resulted also in high-strength reliable bonding to pure titanium.[15]

No data have been published yet on the retention of zirconia ceramic copings to titanium abutments. Therefore, the purpose of this study was to evaluate the effect of 2 surface conditioning methods and 2 luting-gap sizes on the retention and durability of zirconia ceramic copings bonded to titanium abutments.

MATERIALS AND METHODS

Industrially manufactured yttrium oxide partially stabilized zirconia ceramic copings and titanium abutments modified for the test setup (Camlog Biotechnologies), with a luting gap of either 30 µm or 60 µm (Fig 1). The copings and abutments were bonded with the dual-curing composite resin cement Panavia F Ex (opaque white color; Kuraray). For all groups, the bonding surfaces of the titanium abutments were air-abraded with 50-µm aluminum oxide (Al_2O_3) particles at 2.5 bars pressure (0.25 MPa) for 20 seconds at a distance of 10 mm and then were cleaned ultrasonically in 96% isopropyl alcohol for 3 minutes. The bonding surfaces of the ceramic copings were either *(a)* ultrasonically cleaned in 96% isopropyl alcohol for 3 minutes or *(b)* air-abraded with 50-µm Al_2O_3 particles as described and then ultrasonically cleaned.

Thus, 2 luting-gap sizes and 2 methods of surface conditioning were tested. The following codes were assigned to the groups:

- **ALC30:** The luting-gap size was 30 µm. The bonding surface of the titanium abutment was air-abraded, then the zirconia coping and titanium abutment were ultrasonically cleaned in 96% isopropyl alcohol for 3 minutes.
- **ALC60:** The luting-gap size was 60 µm. The bonding surface of the titanium abutment was air-abraded, and the zirconia coping and titanium abutment were ultrasonically cleaned in 96% isopropyl alcohol for 3 minutes.

Fig 1 Industrially manufactured titanium abutments modified for the test setup and the industrially manufactured zirconia ceramic copings. The height of the titanium abutments was 2.7 mm. Abutment diameter was 2.86 mm in groups ALC30 and ABR30 and 2.80 mm in groups ALC60 and ABR60.

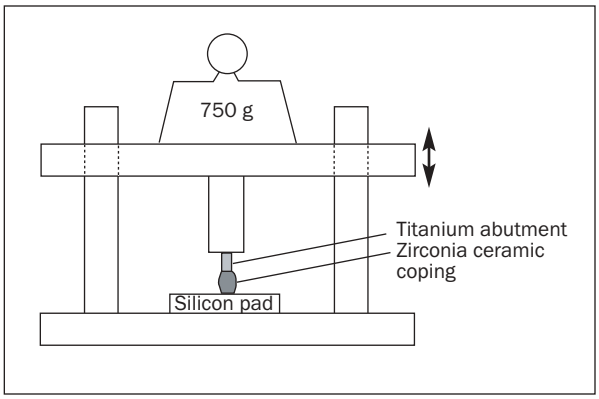

Fig 2 Alignment apparatus for bonding.

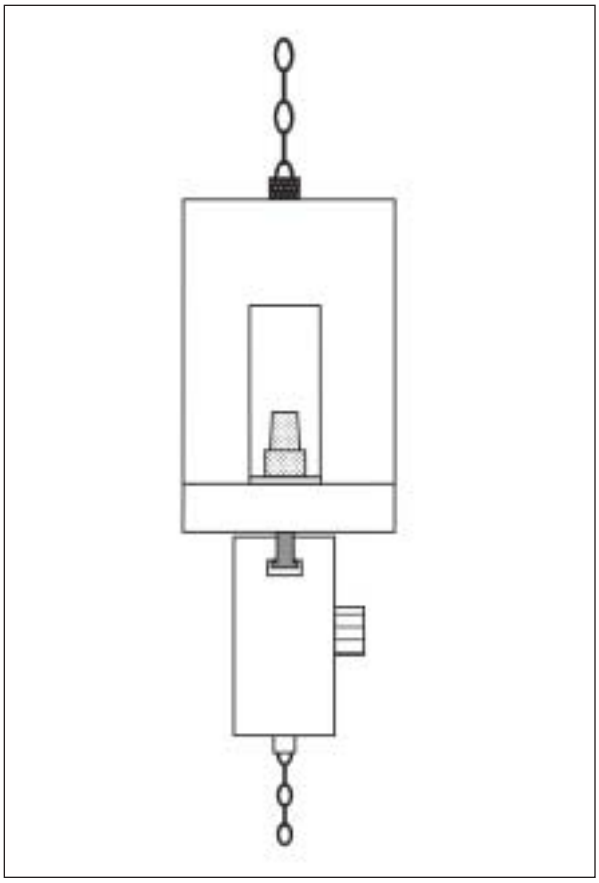

Fig 3 Self-aligning debonding jig: bonded zirconia coping and titanium abutment attached to the load cell with upper and lower chains.

- **ABR30:** The luting-gap size was 30 µm. The bonding surfaces of the titanium abutment and the zirconia coping were air-abraded, then both parts were cleaned in 96% isopropyl alcohol for 3 minutes.
- **ABR60:** The luting-gap size was 60 µm. The bonding surfaces of the titanium abutment and the zirconia coping were air-abraded, then both parts were cleaned in 96% isopropyl alcohol for 3 minutes.

All specimens were bonded using the composite resin cement Panavia F Ex. Within 8 minutes of the start of mixing the composite resin, 2 specimens were bonded at a time using an alignment apparatus that applied a weight of 750 g to the bonded specimens (Figs 2 and 3). This method has been described in detail previously.[16] Excess resin was removed from the bonding margins using pellets, and an oxygen-blocking gel (Oxyguard II; Kuraray) was applied. All specimens were light-cured for 30 seconds using a dental curing light (Heliomat; Vivadent, Schaan, Liechtenstein) within the alignment apparatus and then further cured in a xenon strobe light-curing unit (Dentacolor XS; Heraeus-Kulzer, Wehrheim, Germany) for an additional 180 seconds.

All specimens were then stored in distilled water (37°C). Subgroups of 8 specimens of each group were stored for either 1, 30, 60, or 150 days. In addition, the latter 3 groups were thermal-cycled 15,000, 30,000, or 75,000 times, respectively, between 5 and 55°C with a dwell time of 30 seconds to stress the bond interface.

Table 2 Retention of Zirconia Ceramic Copings Bonded to Titanium Abutments Using 2 Different Methods of Surface Conditioning and 4 Different Storage Times

	Storage times			
Group	1 d without TC	30 d with 15,000 TC	60 d with 30,000 TC	150 d with 75,000 TC
ALC30	313.8 (60.2)	360.3 (40.1)	327.2 (70.9)	411.5 (99.7)
ALC60	230.4 (43.3)	380.6 (122.2)	292.0 (75.3)	350.3 (139.9)
ABR30	874.2 (81.9)	1077.4 (125.1)	1182.6 (130.5)	1107.4 (146.5)
ABR60	688.5 (63.1)	861.1 (206.6)	932.1 (248.1)	871.9 (200.8)

Mean retention values shown in newtons, with standard deviations in parentheses.
ALC = ultrasonically cleaned in alcohol; ABR = airborne-particle abraded and then cleaned in alcohol; 30 = 30-µm luting gap; 60 = 60-µm luting gap; TC = thermal cycles.

Table 3 Summary of the 3-way ANOVA for All Test Groups

Source	SS	DF	MS	F	*P*
Storage time	563463.12	3	187821.04	11.10	.001
Pretreatment	12132109.94	1	12132109.94	716.67	.001
Luting-gap size	545255.14	1	545255.14	32.21	.001
Storage time*Pretreatment	233268.21	3	77756.07	4.59	.050
Storage time*Luting-gap size	13266.29	3	4422.10	0.26	.853
Pretreatment*Luting-gap size	262798.00	1	262798.00	15.52	.001
Storage time*Pretreatment*Luting-gap size	20310.13	3	6770.04	0.40	.753
Error	1895982.16	112	16928.41		
Total	68280324.82	128			

SS = sum of squares; DF = degree of freedom; MS = mean squares; F = variance ratio.

Following storage, the bond strength test was performed. Tension was applied at a crosshead speed of 2 mm/min (Z010/024; Zwick, Ulm, Germany) using a special test configuration that provided moment-free axial force application.[16] A collet held the zirconia abutment while an alignment jig allowed self-centering of the sample. Attachment of the jig to the load cell and crosshead was achieved by upper and lower chains, which further enabled self-alignment of the system.

The fractured interfaces on the titanium surfaces were examined using an optical microscope (Zeiss, Jena, Germany) at a magnification of 30× to calculate the debonded area. A failure mode of either "adhesive" or "cohesive" was assigned. Representative zirconia copings as well as a zirconia coping that did not undergo any treatment and an air-abraded zirconia coping were sectioned and further examined in a scanning electron microscope (SEM; Philips XL 30 CP, Eindhoven, The Netherlands) with an acceleration voltage of 10 kV after sputtering using a gold alloy conductive layer of approximately 30 nm.

Statistical Analysis

The statistical analysis was performed using the 3-way analysis of variance (ANOVA) followed by 2-way ANOVAs depicting the 2 different methods of pretreatment. Results were considered significant where *P* was less than .05.

RESULTS

The mean retention values and standard deviations are summarized in Table 2 for all 4 groups and storage conditions. Three-way ANOVA revealed a significant influence ($P \leq .05$) of the storage condition, the surface-conditioning method, and the luting-gap size on retention (Table 3). Moreover, significant interactions were found between *(a)* the storage time and the surface conditioning method and *(b)* the surface conditioning method and the luting-gap size. Therefore, 2 separate 2-way ANOVAs were performed to evaluate the effect of storage time and luting-gap size for the 2 different methods of surface conditioning (air abrasion with cleaning versus cleaning only) (Tables 4 and 5). There were no significant interactions between the storage time and the luting-gap size for either surface treatment group. However again, the main effects tested (ie, luting-gap size and storage time) were found to be significant, except for the luting-gap size in the non–air-abraded groups.

The mean retention of the non–air-abraded groups was initially relatively low (230.4 N for the ALC30 group and 313.8 N for the ALC60 group); it increased slightly over the different storage times ($P = .076$). However, there were significant differences between the different luting-gap sizes, especially for the 1-day data ($P = .002$). Only the luting-gap size had a statistically significant influence on retention when the specimens had not been air-abraded.

Table 4 Summary of 2-way ANOVA for the Non–air-abraded Groups

Source	SS	DF	MS	F	*P*
Storage time	127285.87	3	42428.62	5.45	.002
Luting-gap size	25487.32	1	25487.32	3.28	.076
Storage time*Luting-gap size	24005.67	3	8001.89	1.03	.837
Error	435718.24	56	7780.68		
Total	7720546.32	64			

SS = sum of squares; DF = degree of freedom; MS = mean squares; F = variance ratio.

Table 5 Summary of the 2-way ANOVA for the Air-abraded Groups

Source	SS	DF	MS	F	*P*
Storage time	669445.45	3	223148.49	8.56	.001
Luting-gap size	782565.81	1	782565.81	30.01	.001
Storage time*Luting-gap size	9570.75	3	3190.25	0.12	.947
Error	1460263.92	56	7780.68		
Total	60559778.50	64			

SS = sum of squares; DF = degree of freedom; MS = mean squares; F = variance ratio.

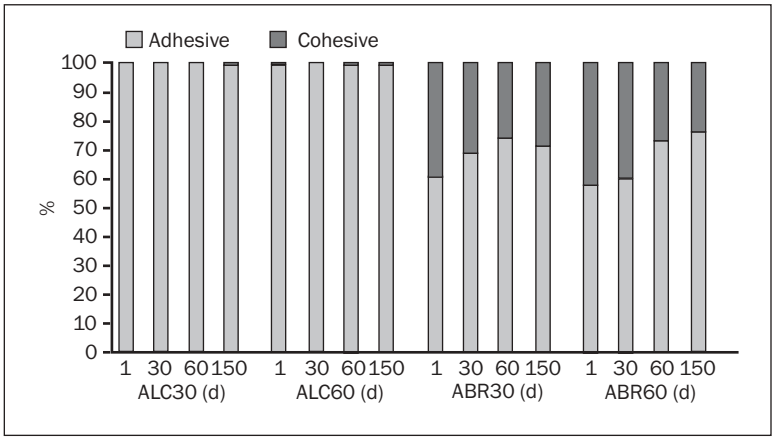

Fig 4 Fracture failure modes as observed using an optical microscope at 30× magnification, calculated as a percentage of the debonded area for all groups after all storage conditions.

The mean retention of the air-abraded groups was much higher and ranged from 688.5 N to 1182.6 N. It increased significantly from 1 day to 60 days for both luting-gap sizes ($P = .001$) but decreased slightly after 150 days. The 60-µm luting gap showed significantly lower retention values than the 30-µm luting gap ($P = .001$).

Overall, airborne particle abrasion led to a statistically significant increase of the retention, whereas the storage time in combination with thermal cycling led to a significant increase of the retention in groups ABR30 and ABR60 between 1 and 150 days of storage.

The failure modes as assigned using the optical microscope at a magnification of 30× and calculated in percentages of the bonding areas are shown in Fig 4 for all groups. In the groups cleaned with alcohol only (ALC30 and ALC60), failure modes were nearly all adhesive at the zirconia ceramic surface for all storage conditions (Fig 5). In the air-abraded groups, ABR30 and ABR60, failure modes were mixed but were still predominantly adhesive at the zirconia ceramic surface.

Representative zirconia copings from each group after 1 day and after 150 days of storage time were sectioned and further examined using an SEM. No differences from the results observed with the optical microscope could be found. Furthermore, no differences in failure mode between the different storage times could be found. In the ALC groups the surfaces were similar to the untreated surface of the industrially manufactured zirconia ceramic coping. In the ABR groups either the surface was similar to the air-abraded zirconia ceramic surface or, in the case of mixed failures, composite resin cement could be observed in places (Fig 6).

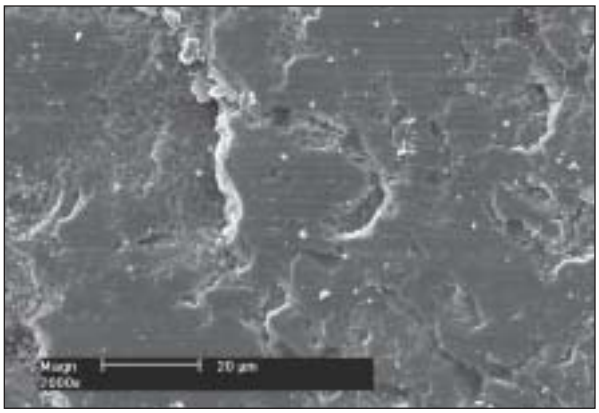

Fig 5 Adhesive failure mode in group ALC30 after 150 days of water storage and thermal cycles. No remnants of the luting composite resin are visible. SEM photograph (original magnification ×2,000).

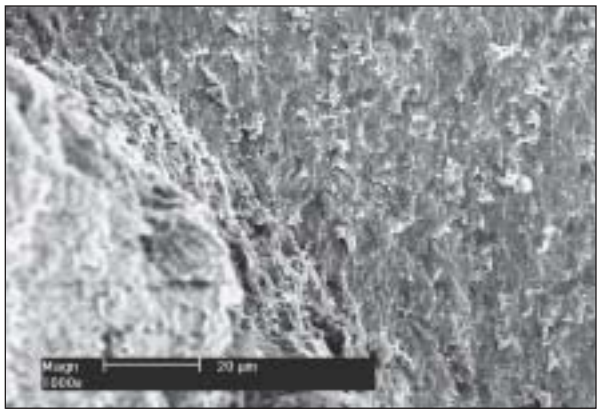

Fig 6 Mixed failure mode in group ABR60 after 1 day of water storage without thermal cycles. Parts of the composite resin layer are visible on the ceramic surface. SEM photograph (original magnification ×2,000).

DISCUSSION

Long-term water storage at a constant temperature and long-term thermal cycling are often used to simulate aging of resin bonds. However, it has been shown that different bonding systems are influenced differently by these 2 parameters.[17] As both are considered clinically relevant aging parameters, in this study long-term water storage was combined with thermal cycling at different intervals to test the long-term durability of the retention of industrial manufactured yttrium-oxide partially stabilized zirconia ceramic copings bonded to titanium abutments using a phosphate monomer-containing composite resin.[17–19]

As the results of this study show, it is possible to achieve good, stable retention between zirconia ceramic copings and titanium abutments using composite resin when airborne particle abrasion is used as a pretreatment. Previous studies using Panavia F and Panavia 21 to bond either zirconia ceramic or titanium have also shown bonding stability.[10,20]

Problems have occurred with the use of zirconia in orthopedic applications (ie, hip implants). These problems have been related to degradation at low temperatures associated with the roughening of the implant after steam sterilization. The roughening of the bonding surface of the zirconia ceramic coping by airborne particle abrasion, which led to an increase of retention, may also affect the long-term integrity of the material itself.[9] No data on the long-term behavior of air-abraded zirconia ceramic in dental restorations is available yet. However, as the resin bond to air-abraded zirconia ceramic specimens was stable over 2 years of artificial aging with no degradation,[13] it is assumed that the resin luting materials have the ability to seal the roughened surface and to prevent negative effects of surface alteration.

Furthermore, the results of this study show that the luting-gap size of 30 µm increased the retention in combination with airborne particle abrasion. The effect of luting-gap size has been debated in the literature and seems strongly dependent on the configuration of the test. The geometry and material of the bonded specimen have differed widely across study designs. For example, a previous study in which 2 plain pieces of glass were bonded together recommended for Panavia 21 a luting-gap size of 10 µm.[21] In contrast, a study[22] in which metal cylinders with various luting-gap sizes between 20 µm and 200 µm were used with storage conditions comparable to those used in the current study found the highest bond strengths with a luting-gap size of 80 µm.

However, other parameters, such as the total bonding surface and the height and width of the abutments affect abutment retention. Studies have shown that the relationship between the height and width of the abutment is more important than the total bonding surface area and that the chimney height of the bonded cylinder significantly affects the failure of the cements.[23,24] As these parameters were not tested in the current study, the results of the present study can be transferred to other implant systems with different geometries only to a limited extent.

Airborne particle abrasion has been recommended as the best method of pretreatment in previous studies to improve the bond strength to oxide ceramics.[14,20,25] Water storage resulted in a significant increase of the retention in the air-abraded groups for up to 150 days. As shown in Table 2, the retention values of the air-abraded groups ABR30 and ABR60 increased over time, reached their highest values at 60 days, and decreased slightly after 150 days of storage. The initial increase in retention

might be explained by postpolymerization effects of water storage, such as stress relaxation, plasticization of the resin matrix, and hygroscopic expansion of the composite resin inside the luting gap due to water uptake.[26,27] The slight long-term decrease of retention between 60 and 150 days in the air-abraded groups might be related to a certain degradation within the composite resin itself, which has previously been shown for other composite resins.[20,28,29] In addition, in the air-abraded groups, the standard deviations increased over storage time, especially in the 60-µm luting-gap group. It is assumed that the resin in the larger gap has a greater variation in air bubbles and voids due to the manual mixing procedure and that the hydrolytic effect caused by water uptake varies with the amount of air bubbles and voids in the resin.

The failure modes of the ALC30 and ALC60 groups were nearly completely adhesive at the zirconia surface. Therefore, in these groups, adhesion to the zirconia ceramic was the weakest link, and the slight degradation within the composite resin had no effect on the retention. Air bubbles and voids in the resin after manual mixing may have affected the composite resin in these groups as well. In contrast, the failure modes of the air-abraded groups (ABR30 and ABR60) had a substantial cohesive portion (Fig 4), which resulted in a higher retention. Also a previous study showed that higher retention correlated with a higher percentage of cohesive failures.[30]

Based on the results of this study, the use of composite resin in combination with air abrasion can be recommended as a method to condition surfaces before bonding zirconia ceramic copings to titanium abutments. For clinical use, a luting-gap size of 30 µm should be preferred over a luting-gap size of 60 µm.

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