

Comparative Evaluation of the Retentive Bond Strength of Air - Abraded Zirconia Copings to Prepared Extracted Human Teeth Using Different Luting Agents – An In Vitro Study”

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Abstract

Aim: To evaluate the retentive bond strength of single unit air abraded Zirconia copings to prepared extracted human teeth cemented with 3 different luting cements.

Material and Methods: Thirty extracted human mandibular first premolar teeth that were prepared to a depth of 1.5 mm, with a 50 taper angle from a vertical axis to create an angle of convergence of 100. The specimens were randomly distributed into two equal groups (n=15): Group A(control), Group B(sandblasting) and subgroups for cementation with the 3 resin-based cements: Multilink® Automix[Ivoclarvivadent], RelyX U200[3M ESPE], Panavia F2.0[Kuraray]). To test the retention of specimens, a universal testing machine was used (0.5mm/minute). Statistical analyses of the data were performed by using one-way analysis of variance (ANOVA) ($\alpha=0.05$).

Result: The mean (SD) coping removal stresses for group A subgroups (N) were control+ Rely X U200 216.40 (90.27), control+ Multilink Automix 336.40 (189.11) and control+ Panavia F2.0 462 (575.69). For group B sandblast+ Rely X U200 412.20 (495.32), sandblast+ Multilink Automix 755.20 (634.19) and sandblast+ Panavia F 2.0 644.60 (495.44), respectively.

Conclusion: Within the limitations of this study, Group BII specimens showed the maximum tensile bond strength scores and proved to be the best option for surface treatment of Zirconia copings.

Keywords: CAD/CAM, Surface Treatment, Zirconia, Sandblasting, Resin Cements

INTRODUCTION

Concerned about the esthetics and biocompatibility of final restorations, dentists have begun demanding metal-free dental restorations. Primarily, because of their reduced physical properties, all-ceramic restorations have been limited to crowns in anterior teeth¹. To overcome this problem, high-strength ceramics such as alumina were developed. Consequently, Zirconia, a high-strength ceramic was introduced for dental applications. This ceramic has several properties making it the material of choice where esthetic and high functional demands are concerned. Because of its high fracture strength, its biocompatibility and its hard and dense surface, Zirconia was recommended for use in posterior restorations²⁻³.

Zirconia is a crystalline dioxide of Zirconium. Its mechanical properties are very similar to those of metals and its color is similar to tooth color. With increasing demand in esthetics and biocompatibility, all-ceramic restorations have gained popularity in recent decade. Among all ceramic systems available, ZIRCONIA OXIDE ceramic has emerged as an excellent esthetic material for fabrication of crowns. The properties of zirconium oxide ceramics such as high strength, excellent mechanical properties and biocompatibility allow it to be used as a core material for all-ceramic crowns and fixed partial dentures (FPDs)⁴. The most utilized Zirconia in dentistry is yttria-containing tetragonal Zirconia polycrystalline⁵⁻⁹.

Zirconia has mechanical properties similar to those of stainless steel. Its resistance to traction can be as high as 900-1200 MPa and its compression resistance is about 2000 MPa. Surface treatments, mechanically or chemically can modify the physical properties of zirconia. ZrO₂ is essentially an inert and nonpolar material, and, in spite of its superiority in terms of mechanical performance, there are some inherent problems, including the adhesion to a variety of substrates¹⁰. For example, acid etchants such as hydrofluoric acid or hydrophosphoric acid do not adequately roughen the surface for micromechanical retention¹¹⁻¹². Therefore, alternative methods have been explored

to bond ZrO₂ such as surface grinding using silicon carbide or aluminium oxide (Al₂O₃) particle air-abrasion or using a diamond bur. This method creates high surface energy, promotes microretention and removes any contaminants from the ceramic surface and also is generally easy to apply⁶⁻⁹

Some studies evaluated different resin-based luting cements with different phosphate monomer containing for bonding to Zirconia. However, it still remains unclear as to which resin-based luting cement and Zirconia produced the most durable bond strengths. Higher chemical affinity would be attained with the use of resin cements containing phosphate monomers, such as 10-methacryloyloxydecyl dihydrogen phosphate (MDP), promoting higher bond strength¹³, or by using additional bond agents, called primers¹⁴⁻¹⁵, which also have these monomers in their composition. The association of resin luting cements with primers promoted a better interaction with ceramic surface due to the increase in cement wetting¹⁵⁻²⁰. This wetting favours the adhesion process and improves the chemical interaction between resin cement and the zirconia surface. These ceramic primers usually contain silane and a functional phosphate monomer. Panavia F2.0 (Kuraray) is composed of the functional monomer 10-MDP, 3-methacryloxypropyltrimetoxysilane (3-MPS) as silane and ethanol. Conventional silane is not effective on zirconia due to the absence of silica in its composition. However, when a silane primer (3-MPS) reacts with 10-MDP, the interaction of the primer with the substrate and resin cement is promoted, forming cross links with the OH groups from ceramic and cement methacrylates. This reaction can be induced and sustained by the acidity of the ceramic treated with the coupling solution.

One of the recently developed phosphate monomers (Rely X U200) has a characteristic of self-etching phosphorylated methacrylates that is designed to bond directly to both enamel and dentine. With two phosphate groups and at least two double bonded carbon atoms, good bond

strength to zirconia plus adequate cross-linking to the resin matrix is achieved.

Another new self-etch phosphate monomer (Multilink Automix) characterized by hydrolytic stability has one phosphate terminal and at least two sites capable of bonding to resin matrix through oxygen bond. This molecule has a terminal hydroxyl group as a substituent that gives the monomer stability under water and in acidic conditions.

In the light of above facts, this study was planned to investigate the retentive bond strength of untreated and air-abraded Zirconia copings bonded to prepared extracted human teeth using three different resin-based luting agents. The Null hypothesis was that there will be no statistically difference in the bond strength of zirconia bonded to prepared untreated human teeth using three different resin-based luting agents.

MATERIAL AND METHODS

Criteria for Selection of Specimen

Thirty extracted human mandibular first premolar teeth that were sacrificed for orthodontic purpose were used. They were cleaned off surface debris, placed in 1% hydrogen peroxide immediately following extraction for 5 minutes and then stored in tap water that was changed weekly till use.

Mounting of Teeth

To retain the specimens in the acrylic blocks during testing, the root surfaces were notched with an inverted cone bur in a high-speed hand-piece. Also, a 0.7 mm diameter hard steel wire was looped through a transverse hole drilled near the apex of each root. The root was embedded into a cylinder which were filled with self-polymerized resin (DPI - RR Cold Cure, India) upto 2mm below the mid facial cemento-enamel junction.

Preparation of Teeth

The tooth with its custom-made jig held firmly in the dental surveyor stand base. For the tooth crown preparation, a straight micromotor hand piece was fixed on a laboratory milling machine to ensure the same preparation angle for each specimen and the cylinder with the tooth was held securely vertically and firmly in a surveyor base. The occlusal surface of each mounted tooth was prepared flat 3 mm

above the top of the cylinders, using a diamond wheel shape bur in a high-speed hand-piece. Using a carbide bur mounted to the milling machine, the axial wall of the teeth were prepared to a depth of 1.5 mm, with a 50 taper angle from a vertical axis. A new rotary instrument was used for each tooth. The resultant preparation had an axial length (occluso-gingivally) of 3 mm with a modified chamfer finish line. All the axio-occlusal line angles of each tooth were rounded. Using a caliper, the prepared teeth were measured mesiodistally (MD) and buccolingually (BL) to minimize the effect of variations in the preparation procedure, the same clinician prepared all specimens.

Fabrication OF ZrO₂ Copings

A customized special tray was made for each prepared tooth using a visible-light polymerized acrylic resin. A special tray adhesive was applied to each custom tray. An impression of each tooth was made with Addition silicone impression material using the respective custom tray. After the impression had set, the trays were removed and the impressions were then poured with type IV gypsum stone. The master die was recovered from the impression, sectioned and trimmed, and a die hardener material was applied.

Thirty Zirconia copings were manufactured using computer-aided design/computer-assisted manufacturing for all prepared teeth. They were distributed into 2 groups of 15 each as follows:

Group A – Untreated copings

Group B – Internal surfaces treated with 50µm Al₂O₃ for 15 seconds at a pressure of 1.5 bars.

Each Group was divided into 3 subgroups for cementation with the 3 resin-based cements.

Cementation of the Zirconium Copings

For Rely X U200, prior to cementation, the prepared tooth was cleaned thoroughly with a water spray. The tooth surface was cleaned. The clicker dispenser was depressed to dispense equal volumes of cement pastes on to the mixing pad. The pastes were mixed using a plastic cement spatula for 20 sec until a uniform color was achieved. A thin layer of cement was applied to the inside surface of each coping. The coping was seated firmly. The margins were light polymerized for 2 sec and excess cement

was removed. Light polymerization was then applied for 20 seconds for each surface.

For Panavia F2.0, prior to cementation, the prepared tooth was cleaned with water spray and dried it before cementation. Equal amounts of ED PRIMER II A& B were mixed and applied to the tooth. After a wait of 30 seconds, tooth was gently air dried. Equal amount of paste A & B were dispensed and mixed for 20 sec. The mixture of the paste was applied to internal surfaces of the copings. Excess cement was removed after tack cure of 2-3 sec with conventional halogen light. Margins were then light cured for 20 sec. per surface (conventional halogen light). Self cure material OXYGUARD II, was applied to the margin and left for 3 min during the self curing process.

For Multilink Automix, prior to cementation, the prepared tooth was cleaned with water spray and dried before cementation. Equal amount of Primer A & B were mixed and applied to the tooth and light cured for 20 sec. This was followed by application of zirconia primer, Monobond Plus on the internal surfaces of copings and dried for 3-5

seconds with an air syringe. With the help of automix tip, paste was dispensed in the internal surfaces of copings and placed on the tooth. Excess cement was removed and margins were light cured for 60 sec per surface.

Retention Test of the Zirconium-Oxide Coping

To test the retention of specimens, a universal testing machine was used. A specially customized chain was made to ensure even distribution of pulling tensile forces using a locking mechanism. The cemented crowns were pulled off along the path of insertion with a crosshead speed 0.5mm/minute. The forces required for dislodgement of the crowns were recorded in N.

Statistical analyses of the data were performed by using A one-way analysis of variance (ANOVA) was applied to the mean retentive bond strengths of different cement materials. When a significant cross product interaction was found, a Tukey multiple comparison test was performed to determine which groups were significantly different. All statistical analyses were performed at a 0.05 level of significance ($\alpha=0.05$).

RESULTS AND OBSERVATIONS

Table 1: Descriptives

Group_2		N	Mean	Std. Deviation	95% Confidence Interval for Mean		P-Value	
					Lower Bound	Upper Bound		
Group A	Surface Area (mm ²)	Group AI	5	23.3600	3.54937	18.9529	27.7671	.035
		Group AII	5	20.5800	1.37868	18.8681	22.2919	
		Group AIII	5	18.3400	2.55768	15.1642	21.5158	
		Total	15	20.7600	3.24484	18.9631	22.5569	
	Test (N)	Group AI	5	216.40	90.268	104.32	328.48	.563
		Group AII	5	336.40	189.111	101.59	571.21	
		Group AIII	5	462.00	575.689	-252.81	1176.81	
		Total	15	338.27	343.526	148.03	528.50	
Group B	Surface Area (mm ²)	Group BI	5	21.6560	2.67875	18.3299	24.9821	.712
		Group BII	5	20.7300	3.19367	16.7645	24.6955	
		Group BIII	5	20.2700	2.01358	17.7698	22.7702	
		Total	15	20.8853	2.54533	19.4758	22.2949	
	Test (N)	Group BI	5	412.20	495.318	-202.82	1027.22	.571

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The mean (SD) coping removal stresses for group A subgroups (N) were control+ Rely X U200 216.40 (90.27), control+ Multilink Automix 336.40 (189.11) and control+ Panavia F2.0 462 (575.69). Control+ Panavia F2.0 showed the highest mean crown removal stress; however, because one coping fractured during the test. Followed by control+ Multilink Automix than Control+ Rely X U200 found lowest removal stress in this group. For all above-mentioned groups, the mean dislodgement

stress was influenced by the cohesive strength of the tooth and the cohesive stress of the zirconia coping. The mean (SD) coping removal stresses (N) for group B sandblast+ Rely X U200 412.20 (495.32), sandblast+ Multilink Automix 755.20 (634.19) and sandblast+ Panavia F 2.0 644.60 (495.44), respectively. Multilink Automix exhibited the highest mean crown removal stress followed by Panavia F 2.0 and Rely X U200.

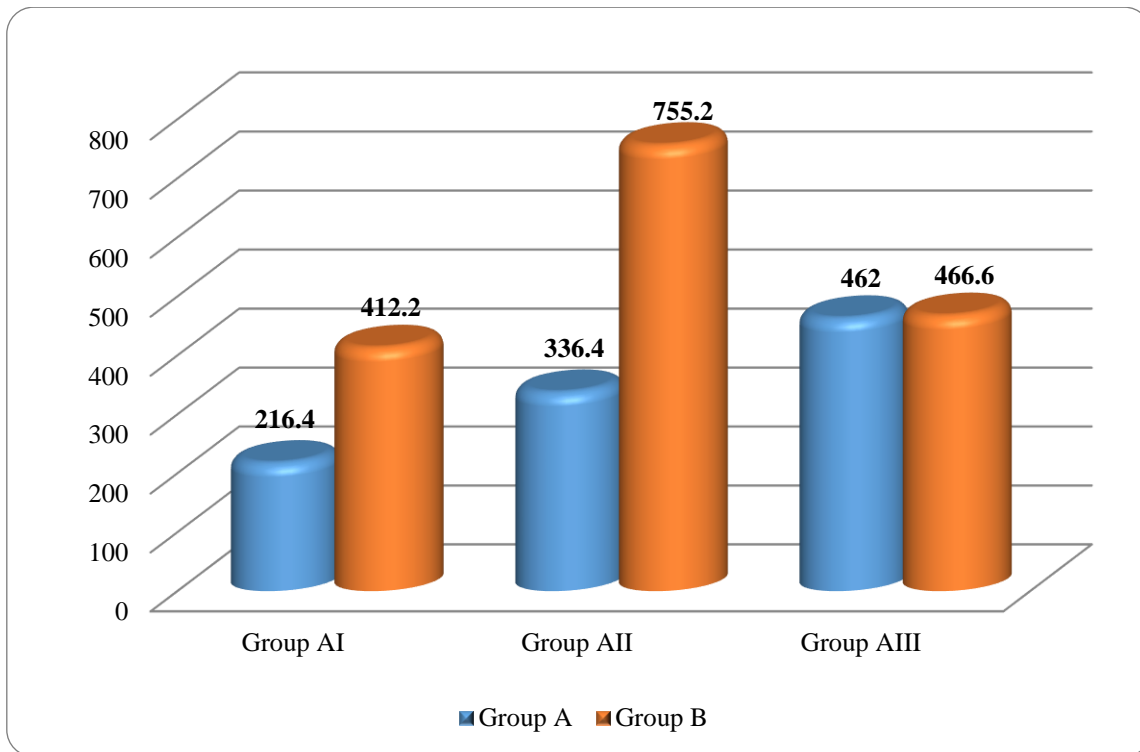
Table 2: Multiple Comparisons (LSD)

Group 2	Dependent Variable	(I) Group 1	(J) Group 1	Mean Difference (I-J)	Std. Error	P-Value	95% Confidence Interval	
							Lower Bound	Upper Bound
Group A	Surface Area (mm ²)	Group AI	Group AII	2.78000	1.67493	.123	-.8694	6.4294
			Group AIII	5.02000*	1.67493	.011	1.3706	8.6694
		Group AII	Group AIII	2.24000	1.67493	.206	-1.4094	5.8894
	Test (N)	Group AI	Group AII	-120.000	223.705	.601	-607.41	367.41
			Group AIII	-245.600	223.705	.294	-733.01	241.81
		Group AII	Group AIII	-125.600	223.705	.585	-613.01	361.81
Group B	Surface Area (mm ²)	Group BI	Group BII	.92600	1.69035	.594	-2.7570	4.6090
			Group BIII	1.38600	1.69035	.428	-2.2970	5.0690
		Group BII	Group BIII	.46000	1.69035	.790	-3.2230	4.1430
	Test (N)	Group BI	Group BII	-343.000	323.004	.309	-1046.76	360.76
			Group BIII	-232.400	323.004	.486	-936.16	471.36
		Group BII	Group BIII	110.600	323.004	.738	-593.16	814.36
*. The mean difference is significant at the 0.05 level.								

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From the ANOVA results of the above table, comparison within the groups was not statistically significant with respect to retentive bond strength

i.e. higher than 0.05 at 5% level of significance. It means that, the retentive bond strength scores are different in two groups (group AI, AII, AIII, and group BI, BII, BIII).



Graph 1: Mean Values of Test Groups

One-way ANOVA was first applied to these data, because two categorical factors (surface treatment and cement type) are associated with a continuous outcome (coping removal stress), the outcomes are not related to each other, and the shape of the histogram was not statistically significantly different from the normal curve; however, the assumption of equal variance was not violated. In Graph, Rely X U200 was having lowest mean retentive strength values in both the groups whereas, Panavia F2.0 having highest retentive values in Group A and in Group B Multilink Automix having highest mean retentive values.

The results for characterization of failure type are presented. Overall, the predominant mode of failure for Group A I 3 of the specimens had cement in the copings followed by 2 of the specimens with cement principally on the tooth. In contrast, failure modes for Group A II were 4 for cement principally on the coping, 1 with cement principally on the tooth. The group of copings cemented with Group A III 2 of the specimens had cement in the copings followed by 2 of the specimens with cement principally on the tooth, and 1 where tooth or root fracture. The predominant mode of failure for

group B I 3 of the specimens had cement on the tooth, 1 on the coping and 1 tooth or root fractured. Group B II mode of failure 1 of the specimens had cement on the coping, 3 within the tooth and 1 root or tooth fracture. For Group B III had 3 of the specimens with cement on the coping, 1 with the cement on the tooth and 1 with the root or tooth fracture.

DISCUSSION

The present study revealed that retention of copings/crowns depends on the following factors: preparation design of the prepared tooth surface, any surface treatments given on the intaglio surface of the copings/ crowns, type of resin cements used. In this study, Zirconia oxide blanks (ZrO₂ stabilized by Y₂O₃) from Ziecon, were used to prepare the copings by CAD/CAM on the prepared extracted human mandibular premolar teeth with 3mm axial length and 5 degrees of taper as per previous in-vitro studies by Khalil Aleisaet al¹⁰. The samples were divided into 2 groups, one as the control and the other with copings being sandblasted before cementation. Each group was further divided into 3 sub-groups for luting the copings with the 3 selected resin cements with

different phosphate monomer chemistry, viz. Rely X- U200, Multilink Automix and Panavia F2.0

Several studies by Markus B. Blatz et al, Gokhan Akgungor et al, Jeong-yeon Yun et al^{14,19,20} have established airborne particle abrasion is considered to be the most effective method for treating zirconia ceramics by improving surface roughness and creating micro-mechanical interlock with the luting agent. Airborne particle abrasion also cleans and increases the surface area, resulting in higher bond strength due to mechanical retention. In our study, in the experimental group, the Zirconia copings were sandblasted using 50um Al₂O₃ at 15 bars pressure for 15 seconds.

However, there have been some concerns raised by Mona W et al²² regarding possibility of micro-cracks formation at the inter-grain level which could affect the longevity of the ceramic restoration. On the other hand, there are studies indicating that air abrasion might even strengthen zirconia ceramics when done cautiously as regards the time to which the specimens are subjected to sandblasting and particle size of the Al₂O₃.²¹

In our study, we have used 3 resin based luting cements based on different chemistry related to the phosphate monomers used. Rely X U200 dispenser is a dual-cure, two-paste, hand mix resin material containing methacrylate monomers with phosphoric acid groups. This cement is able to make a hydrogen bond with the zirconia surface because the phosphoric acid groups in its composition promote this surface bonding.

Multilink Automix is a dual-cure, two-paste, automix resin material containing phosphate monomer characterized by hydrolytic stability, has one phosphate terminal and at least two sites capable of bonding to resin matrix through oxygen bond. This molecule has a terminal hydroxyl group as a substituent that gives the monomer stability under water and in acidic conditions.

Panavia F 2.0 is a dual-cure, two-paste, hand mix resin material containing functional monomer 10-MDP (10-methacryloyloxydecyl dihydrogen phosphate), 3-methacryloyloxypropyltrimethoxysilane (3-MPS) as silane and ethanol. Conventional silane is not effective on zirconia due to the absence of

silica in its composition. However, when a silane primer (3-MPS) reacts with 10-MDP, the interaction of the primer with the substrate and resin cement is promoted, forming cross links with the OH groups from ceramic and cement methacrylates. This reaction can be induced and sustained by the acidity of the ceramic treated with the coupling solution.

Taking the probability Type I Error (α) = 0.05 & Power (1- β) = 0.8, no. of groups in this study being 3 and the effect size (largest difference between any 2 mean divided by SD) was derived as 2.73. Accordingly, as per calculation, the sample size (n) was arrived as 3 per group. For sake of convenience and not to lose any precision in the study, we took the sample size (n) as 5. The results of this study showed no statistically significant difference in the bond strength values among the three groups tested and hence, the Null Hypothesis was accepted.

The results showed that in Group A (untreated samples), the mean surface area of the prepared teeth were not significantly different across the 3 sub-groups. The mean retentive bond strength values also showed no statistically significant difference across the subgroups. Specimens cemented with Rely-X U200 showed the least values (216.40N) and Panavia F2.0 showed the highest (462N). It was interesting to note that the specimens cemented with Panavia F2.0 showed the highest values in spite of them having the least surface area of the prepared teeth. In Group B (sand blasted samples), the mean surface area of the prepared teeth were not significantly different across the 3 sub-groups. The mean retentive bond strength values also showed no statistically significant difference across the subgroups. Specimens cemented with Rely-X U200 showed the least values (412.20N) and Multilink Automix showed the highest (755.20N). It is evident that sandblasting the copings has a positive effect on the retentive values among all 3 sub-groups. It is interesting to note that sandblasting had the most positive effect on the retentive bond strength values of the samples cemented with Multilink Automix. In Group B, all the sub groups showed fracture of

the tooth/root and dislodgement of the tooth itself from the resin block, in 1 sample each before debonding of the coping. This was due to the high bond strength values observed in Group B across all the 3 sub-groups.

Studies done by Clayton GH et al²³ and Sheets JLet al²⁴ have shown that, dislodging loads in natural tooth intra-orally range between 207-509N. From our study, it may be inferred that all the three cements may be capable of retaining the ZrO₂ copings successfully, with and without treatment air-braded of the internal surface of each coping.

The findings of this study were in accordance with the study by Palacios et al¹⁸, in which no statistically significant difference between three different resin types was found; however, in their study, the retention values for Panavia F 2.0 and RXU were higher than the retention values recorded in this study. The possible explanation could be that the zirconia copings tested in that study were different in manufacturing system; therefore, conclusion drawn for one zirconia system may not be valid for others.

In another study, Kern and Wegner²⁵ airborne-particle abraded the zirconia ceramic surface with 110-µm aluminum oxide, applied different luting agents, and found that Panavia F 2.0 provided the highest bond strength values. This is in partial agreement with the results of our study as in group A, the retention value of Panavia F2.0 cement was the highest. In a previous study by Hesam Mirmohammadi et al, they had reported that Multilink Automix showed the highest bond strength after sand blasting where the samples had shown cohesive failure reflecting the capacity of its monomer for bonding to zirconia and tooth surface.

The superior performance of Multilink Automix could be due to its chemistry characterized by hydrolytic stability, having one phosphate terminal and at least two sites capable of bonding to resin matrix through oxygen bond.

Overall, we may say that all the 3 cements tested may show satisfactory clinical performance, both with untreated and treated Zirconia crowns. However, considering the significant increase in retentive values after sandblasting, surface treatment of the intaglio surface of Zirconia crowns by sandblasting can be an easy, practical and useful procedure prior to cementation. This assumes more importance in cases where the retention and resistance forms of the prepared tooth has already been compromised due to any reason. Multilink Automix may be the luting agent of choice in such situations for luting air abraded Zirconia crowns.

The results of this study however, have to be seen in light of some limitations: The samples were stored in water for only 24 hours and were not subjected to thermocycling or fatigue cycle testing and hence could not simulate the complex intra-oral environment before testing.

Within the limitation of this study, the following conclusions can be drawn: there is no statistically significant difference in the bond strength in both the groups under investigation. Panavia F2.0 shows the best bond strength with untreated Zirconia copings while Multilink Automix shows the best bond strength after the copings are sandblasted. Sandblasting the Zirconia based crowns may be made a mandatory procedure prior to cementing them with resin based luting agents to ensure adequate long term clinical performance.

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