COMPARATIVE STUDY OF THE SHEAR BOND STRENGTH OF ZIRCONIA-BASED DENTAL CERAMICS TO RESIN CEMENTS AFTER CHEMO-MECHANICAL SURFACE TREATMENT

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ABSTRACT

INTRODUCTION: The loss of retention of zirconia-based fixed partial dentures (FPDs) is one of the most frequently reported failures.

OBJECTIVES: The purpose of this study was to evaluate the shear bond strength (SBS) of four resin cements to zirconia after air-borne particle abrasion with alumina of different grades and application of respective primers incorporating organophosphate monomers.

MATERIAL AND METHODS: The 443 cuboid-shape zirconia specimens (Ceramill Zi; Amann Girrbach AG) were divided into 16 research groups, according to the chemo-mechanical modification: air-abrasion with alumina particles of 50 µm, 110 µm or 250 µm grade, no treatment and application of primers and resin cements: Clearfil Ceramic Primer/Panavia F2.0 (Kuraray Medical), Monobond Plus/Multilink Automix (Ivoclar Vivadent AG), AZ-Primer/ResiCem (Shofu), Z-Prime Plus/Duo-Link (Bisco). After storage in 37 ± 1°C distilled water for 24 hours, the SBS test was conducted (Zwick/Roell Z005; Zwick GmbH&Co.KG) at 1 mm/min crosshead speed to failure. Specimens were subjected to fractographic analysis at ×2.5 and ×35 magnification (Seliga Auctus; Seliga Microscopes Sp.z o.o.; SEM S-4700; Hitachi High-Technologies Corporation). Statistical significance was established with the Wilcoxon signed-rank test and the Kruskal-Wallis one-way analysis of variance ($p = 0.05$).

RESULTS: The highest SBS values of zirconia to resin cement were obtained for Z-Prime Plus/DuoLink (8.24-14.15 MPa) and after 50 µm alumina air-abrasion (6.87-14.15 MPa). However, the alumina particle grade was insignificant. A significant decrease in the SBS was observed for untreated specimens (4.60-8.24 MPa). The failure mode in untreated specimens was mainly adhesive, while in sandblasted it was cohesive and mixed.

CONCLUSIONS: The chemo-mechanical treatment of zirconia surface before adhesive cementation is crucial to obtain satisfactory values of shear bond strength to resin cements.

KEY WORDS: zirconia, resin cement, shear bond strength, aluminum oxide, air-borne particle abrasion.

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INTRODUCTION

Zirconia-based dental ceramics are widely used in prosthodontics due to their aesthetics, biocompatibility and outstanding mechanical properties. Five-year clinical observations indicate that the survival rate of zirconia-based fixed partial dentures (FPDs) reaches 73.9-100% [14, 17]. The most common failures reported were chipping of the veneering ceramic, loss of retention, framework fractures and secondary caries [14, 17, 29]. Framework fractures and secondary caries resulted mainly from improper handling of the material, such as uneven or too thin framework thickness or improper gingival margin adaptation, whereas the issue of ceramic veneer chipping or loss of retention may be caused by zirconia properties that lead to bonding failures. 3Y-TZP (3 mol% yttria stabilized tetragonal zirconia polycrystals) is a chemically inert material with smooth surface and dense microstructure composed of crystals without silica or any substantial glassy phase [4, 21, 29].

Bonding failure may occur because of inappropriate choice of the luting agent or inappropriate zirconia surface modification before the cementation procedure. Traditional acid-base cements such as zinc-phosphate, zinc-polycarboxylate, glass-ionomer and resin-modified glass-ionomer cements can be used, but resin cements are preferable [6, 11, 23]. They ensure higher bond strengths, low water solubility, better mechanical properties, lower viscosity, better aesthetics, and lower risk of micro-leakage [6, 11, 21]. Self-etch and dual-cure resin cements are recommended for use in the case of zirconia-based ceramic FPDs. However, the adhesive cementation process is complex and requires the proper zirconia surface modification before the application of the bonding agents. The bond strength to resin cements obtained for the other types of ceramics is better than for zirconia-based ceramics [12, 21]. Bonding to zirconia restorations is difficult due to its chemical inertness and microstructure – the crystalline phase without the glassy phase [4, 6, 21, 29]. The proper zirconia surface preparation is then the significant factor improving the strength and durability of the bonding. There are mechanical and chemical types of modifications recommended, i.e. grinding, air-borne particle abrasion with alumina particles, silica coating, selective infiltration etching technique, experimental hot etching solution, laser preparation, MVD (molecular vapor deposition), melted glass micropearl deposition, conventional or experimental primers application, etc. [1, 2, 5, 8, 13, 20, 21, 24]. There are no long-term observations of the influence of these modifications on the properties of zirconia. It is not known, either, which method is the most effective and harmless. The method of choice – effective and easy to perform in every dental office – is air abrasion with alumina particles combined with the use of conventional primers including the 10-MDP monomer (10-methacryloyloxydecyl dihydrogen phosphate) [2, 7, 13, 18, 22, 25, 27, 28].

It is proven that conventional primers containing 10-MDP monomer are more effective in bonding to zirconia-based ceramics than other primers [2, 7, 8, 12, 13, 18, 24, 26, 27]. Phosphate monomers exhibit chemical affinity for zirconium oxide [8, 13, 24]. However, such primer bonding efficacy is satisfactory when combined with air-borne particle abrasion with alumina particles [2, 5, 7, 13, 16, 18, 22, 27, 28]. Omitting air abrasion results in a decrease in bond strength, even bonding failure [2, 5, 16, 18, 22, 25, 27].

There is still no clear algorithm stating the cementation procedure of zirconia-based ceramic FPDs. It is not clearly defined which grade of alumina particle used for air-abrasion is the most effective in improving the bond strength of resin cements to 3Y-TZP, but also the least destructive to its surface, and also which primer along with resin cement ensures the highest bond strength values. The aim of this study was to evaluate the efficacy of air-borne abrasion with alumina particle of different grades and the application of four primers with respect to the shear bond strength of zirconia-based ceramic to resin cements.

OBJECTIVES

The purpose of this study was to evaluate the shear bond strength (SBS) of four resin cements to zirconia after air-borne particle abrasion with alumina particles of different grade and application of respective primers incorporating organophosphate monomers.

MATERIAL AND METHODS

SPECIMEN PREPARATION

The blanks of commercial 3Y-TZP ceramics were used in this study (Ceramill Zi; Amann Girrbach AG, Koblach, Austria). Four hundred and forty-three cuboid-shaped samples with the dimensions of $6.3 \times 6.3 \times 16$ mm were designed and then milled (Ceramill Mind, Ceramill Motion; Amann Girrbach AG, Koblach, Austria). All specimens were sintered for 10 hours (Ceramill Therm; Amann Girrbach AG, Koblach, Austria); the temperature growth was $8^\circ\text{C}/\text{min}$ from 200°C to 1450°C , two hours of constant temperature and then the cooling time. The material shrinkage was approximately 21%. Eventually specimens were $5 \times 5 \times 12.6$ mm in dimension.

The specimens were randomly divided into sixteen groups depending on the grade of alumina particles used for air abrasion and on the type of resin cement and primer used (Table 1). One fourth of samples were left untreated. The others were alumina air-abraded with three different grades of alumina particle, $50\ \mu\text{m}$, $110\ \mu\text{m}$ or $250\ \mu\text{m}$ (Alustral; Omnident Dental Handelsgesellschaft mbH, Rodgau, Germany). All specimens were air-abraded in the same conditions: at 45° angle, at 13 mm distance, at

the pressure of 3 bar, for 10 seconds (Basic Classic Renfert GmbH, Hilzingen, Germany). The surface of each specimen was cleaned with an oil-free air stream. Alumina air-abrasion was followed by the cementation procedure.

Depending on the group division, for each alumina particle grade, four different resin cements with compatible primers were used. Each primer was applied with a microbrush according to the manufacturer's directions. Respective resin cements were layered using a Teflon mold and light-cured (Flashmax P3; CMS Dental, Copenhagen, Denmark). All specimens were stored in distilled water at temperature $37 \pm 1^\circ\text{C}$ for 24 hours.

There were four different resin cements compatible with respective primers containing phosphate monomers used in this study. Their chemical composition disclosed by manufacturers is shown in Tables 2 and 3:

- Primer: Clearfil Ceramic Primer, Cement: Panavia F 2.0 (Kuraray Medical Inc., Sakazu, Kurashiki, Okayama, Japan),
- Primer: Monobond Plus, Cement: Multilink Automix (Ivoclar Vivadent AG, Schaan, Liechtenstein),
- Primer: AZ-Primer, Cement: ResiCem (Shofu Inc., Kyoto, Japan),
- Primer: Z-Prime Plus, Cement: Duo-Link (Bisco Inc., Schaumburg, USA).

SHEAR BOND STRENGTH TESTING

All specimens were subjected to the shear bond strength test using the Universal Testing Machine (Zwick/Roell Z005; Zwick GmbH & Co. KG, Ulm, Germany) at

TABLE 1. Experimental groups

The grade of alumina used for air-borne particle abrasion	Primer and resin cement			
	Clearfil Ceramic Primer + Panavia F 2.0	Monobond Plus + Multilink Automix	AZ Primer + ResiCem	Z-Prime Plus + Duo-Link
–	Panavia 0	Multilink 0	ResiCem 0	Duo-Link 0
50 μm	Panavia 50	Multilink 50	ResiCem 50	Duo-Link 50
110 μm	Panavia 110	Multilink 110	ResiCem 110	Duo-Link 110
250 μm	Panavia 250	Multilink 250	ResiCem 250	Duo-Link 250

TABLE 2. Resin cement chemical composition provided by manufacturers

Resin cement	Main composition provided by the manufacturer	Manufacturer
Panavia F 2.0	Paste A: 10-Methacryloyloxydecyl dihydrogen phosphate (MDP), hydrophobic aromatic dimethacrylate, hydrophobic aliphatic dimethacrylate, hydrophilic aliphatic dimethacrylate, silanated silica filler, silanated colloidal silica, dl-camphorquinone, catalysts, initiators Paste B: hydrophobic aromatic dimethacrylate, hydrophobic aliphatic dimethacrylate, hydrophilic aliphatic dimethacrylate, silanated barium glass filler, surface treated sodium fluoride, catalysts, accelerators, pigments	Kuraray Medical Inc.
Multilink Automix	The monomer matrix is composed of dimethacrylate and hydroxyethylmethacrylate (HEMA). The inorganic fillers include barium glass, ytterbium trifluoride and spheroid mixed oxide.	Ivoclar Vivadent AG
ResiCem	Paste A: UDMA, TEGDMA, fluoroaluminosilicate glass, initiator Paste B: UDMA, TEGDMA, carboxylic acid monomer, fluoroaluminosilicate glass, initiator	Shofu Inc.
Duo-Link	Base: Bis-GMA, TEGDMA, UDMA, glass filler Catalyst: Bis-GMA, TEGDMA, glass filler	Bisco Inc.

UDMA – urethane dimethacrylate, TEGDMA – triethylene glycol dimethacrylate, Bis-GMA – bisphenol A-glycidyl methacrylate

TABLE 3. Chemical composition of primers provided by manufacturers

Primer	Main composition provided by the manufacturer	Manufacturer
Clearfil Ceramic Primer	3-methacryloxypropyltrimethoxysilane, 10-methacryloyloxydecyl dihydrogen phosphate (MDP), ethanol	Kuraray Medical Inc.
Monobond Plus	Alcohol solution of silane methacrylate, phosphoric acid methacrylate, sulfide methacrylate	Ivoclar Vivadent AG
AZ – Primer	Acetone, phosphoric acid monomer	Shofu Inc.
Z – Prime Plus	Biphenyl dimethacrylate, hydroxyethyl methacrylate, ethanol	Bisco Inc.

1 mm/min crosshead speed to failure. To ensure the even distribution of tension during the test, the crosshead was specially designed to match the specimen.

FRACTOGRAPHIC ANALYSIS

All specimens subjected to SBS test were assessed at $\times 2.5$ magnification in order to examine the mode of failure (Seliga Auctus; Seliga Microscopes Sp. z o. o., Lodz, Poland). Cohesive, adhesive or mixed failure modes were observed. Random specimens were also evaluated with an electron microscope at magnification $\times 35$ (Scanning Electron Microscope S-4700; Hitachi High-Technologies Corporation, Tokyo, Japan).

STATISTICAL ANALYSIS

The influence of various modifications of the zirconia surface combining air-borne particle abrasion with alumina particle of different grade and the use of four different resin cements with respective primers on shear bond strength of zirconia-based dental ceramics to resin cements was evaluated with the Wilcoxon signed-rank test and the Kruskal-Wallis one-way analysis of variance, with a level of significance set at 0.05.

RESULTS

The mean shear bond strength values (MPa) and standard deviations of all tested groups are shown in Figure 1 and Table 4. The results of the failure mode assessment are shown in Figure 2, Figure 3 and Table 5. The highest shear bond strength of resin cement to zirconia-based ceramics was obtained for Duo-Link in combination with Z-Prime Plus (8.24-14.15 MPa), the lowest for ResiCem and AZ-Primer (5.26-8.58 MPa). The Wilcoxon signed-rank test ($p = 0.05$) revealed significant differences for Z-Prime Plus + Duo-Link and for AZ-Primer + ResiCem when compared to other resin agents. There were no significant differences between Clearfil Ceramic Primer + Panavia F2.0 and Monobond Plus + Multilink Automix groups. The values obtained for these two resin cements were between those obtained for ResiCem and Duo-Link. In groups in which the alumina air-abrasion was omitted, there were no significant differences between the Panavia 0 group and the ResiCem 0 group.

The highest values of the shear bond strength of zirconia-based ceramics to resin cements were obtained for specimens air-abraded with 50 μm alumina particles, although the Kruskal-Wallis one-way analysis of variance revealed no significant difference between evaluated grades of alumina particles used for air-abrasion ($p = 0.05$). On the other hand, there was a significant difference in the shear bond strength of 3Y-TZP to resin cements between alumina air-abraded specimens and

the specimens in which only primer was used (untreated specimens/control groups). The shear bond strength obtained for specimens without mechanical modification was more than twice as low. The failure mode observed for specimens that were not alumina air-abraded was mainly adhesive, while in sandblasted specimens it was cohesive and mixed.

DISCUSSION

The results obtained in this study indicate that there were significant differences between groups depending on the resin cement and primer used. The highest SBS values were obtained for Duo-Link and Z-Prime Plus (8.24-14.15 MPa, mean 11.61 MPa), independently of the mechanical modification and compared to other resin cements. The explanation for such results was considered in reference to the chemical composition of resin cements and primers used in the current study. There are two monomers present in the primer Z-Prime Plus which have a chemical affinity for 3Y-TZP, i.e. 10-MDP and BPDm (biphenyl dimethacrylate). Such a formula may enhance chemical bonding to zirconia-based ceramics and probably is the reason why application of this primer yielded significantly increased SBS values. Both monomers are reactive to 3Y-TZP, as the MDP compound has two active centers and BPDm has at least two potentially active centers which permit chemical bonding with inert zirconia.

On the other hand, the application of AZ-Primer resulted in decreased SBS values (5.26-8.58 MPa, mean 6.85 MPa). This primer is composed of the phosphoric acid monomer and acetone serving as a solvent. MDP alone without any additional agents may be not efficient enough in yielding increased bonding capacity of zirconia to resin cement. 3Y-TZP has a relatively non-polar nature and its structure is polycrystalline without the presence of silica. Therefore, silane coupling compounds are not effective; besides, they are unstable in the acidic environment [8, 13]. Zirconia-based ceramics are also more stable chemically than the silica containing ceramics, thus they are less prone to water hydrolysis. Also, the Clearfil Ceramic Primer and the Monobond Plus Primer gave lower SBS values. Clearfil Ceramic Primer is composed of solvent, silane coupling compound and MDP agent. It does not incorporate any additional chemical compounds enhancing chemical affinity towards zirconia. Moreover 3-methacryloxypropyltrimethoxysilane may become unstable in the company of acidic MDP. The Monobond Plus Primer acts similarly as the Clearfil Ceramic Primer, as it is composed of silane coupling compounds, phosphoric acid methacrylate and sulfide methacrylate.

Xie *et al.* and Magne *et al.* confirm those theoretical considerations; they indicate that experimental primers based on organophosphate and carboxylic acid monomers

FIGURE 1. Mean shear bond strength values of zirconia to resin cements obtained for 16 experimental groups

TABLE 4. Mean values of shear bond strength of zirconia-based ceramics to resin cement, standard deviations, medians, maximum and minimum values (MPa) for all tested groups

Groups	Shear bond strength (MPa)				
	Mean	Standard deviation	Median	Maximum value	Minimum value
Panavia 0	4.60	2.21	3.66	11.23	1.90
Multilink 0	6.31	2.79	5.57	14.15	3.14
ResiCem 0	5.26	2.58	4.46	12.27	2.11
Duo-Link 0	8.24	3.21	7.09	15.70	4.02
Panavia 50	12.18	3.98	12.01	21.08	5.29
Multilink 50	11.59	3.30	11.59	16.84	7.78
ResiCem 50	6.87	3.39	5.45	13.37	3.24
Duo-Link 50	14.15	5.95	12.70	29.00	7.26
Panavia 110	11.79	3.83	12.32	18.25	6.31
Multilink 110	10.45	3.14	10.35	16.41	4.60
ResiCem 110	6.57	3.07	4.95	16.98	3.90
Duo-Link 110	13.55	4.50	12.90	27.59	7.78
Panavia 250	10.13	3.43	8.95	18.82	6.05
Multilink 250	9.93	2.81	9.21	15.99	5.04
ResiCem 250	8.58	3.36	8.26	16.13	3.25
Duo-Link 250	12.39	4.31	11.36	23.63	5.72

TABLE 5. Percentage of failure modes observed after shear bond strength testing

Experimental groups	Failure mode (%)		
	Adhesive	Mixed	Cohesive
Panavia 0	81	19	0
Multilink 0	39	59	2
ResiCem 0	72	28	0
Duo-Link 0	15	80	5
Panavia 50	10	86	5
Multilink 50	0	95	5
ResiCem 50	10	75	15
Duo-Link 50	0	20	80
Panavia 110	11	82	7
Multilink 110	0	88	12
ResiCem 110	4	85	12
Duo-Link 110	0	63	37
Panavia 250	0	89	11
Multilink 250	4	89	7
ResiCem 250	11	85	4
Duo-Link 250	0	43	57

increase the resin cement bonding capacity to zirconia-based ceramics. Moreover, they prove that 10-MDP has a chemical affinity for 3Y-TZP and the presence

of solvent, such as ethanol, is crucial to create chemical bonds between this compound and zirconia-based ceramics [13, 24].

There are only a few research studies that compare the commercial primers and resin cements used in the current study, but their results are similar. Griffin *et al.* compared the same resin agents and they indicate that the highest SBS values are obtained for Z-Prime Plus and Duo-Link (28.7 MPa), followed by Monobond Plus and Multilink Automix (26.4 MPa), AZ Primer and ResiCem (21.2 MPa), Clearfil Ceramic Primer and Panavia F2.0 (7.5 MPa) [8]. The main difference between the two studies are the results obtained for AZ Primer; however in their study there is a lack of information on the exact air-borne particle abrasion parameters. In the current study, using the air-borne particle abrasion with 250 µm alumina particles resulted in higher SBS values when AZ-Primer was applied on the zirconia surface. El-Shrkawy *et al.* proved that for Multilink Automix resin cement, the additional air-abrasion with 110 µm alumina particles resulted in twice as high bond strength values than for untreated zirconia specimens (13.2 MPa vs. 6.1 MPa) [5]. On the other hand, Kitayama *et al.* compared the bond strength of five different resin cements to zirconia-based ceramics with and without additional primer application; prior to adhesive cementation, they treated the zirconia surface with 70 µm alumina air-abrasion. They found that additional primer application resulted in nearly twice as high bond strength values [12]. Samimi *et al.* assessed the bond strength of zir-

FIGURE 2. Representative SEM micrograph of the failure mode of zirconia surface/resin cement interface at $\times 35$ magnification of specimens treated with air-borne particle abrasion with $50\ \mu\text{m}$ alumina particles. **A)** Panavia F 2.0 and Clearfil Ceramic Primer, **B)** Multilink Automix and Monobond Plus, **C)** ResiCem and AZ-Primer, **D)** Duo-Link and Z-Prime Plus

conia samples to resin cement incorporating the MDP compound (Panavia F 2.0) and to resin cements without organophosphate monomers (Aegis and Esthetic); all samples were alumina air-abraded prior to the cementation procedure. The use of the resin cement incorporating MDP compound resulted in obtaining almost three times higher bond strength values in comparison with resin cements without MDP compound in their composition. Moreover, the use of additional air-borne particle abrasion on the zirconia surface is important as it gave significantly higher bond strength values for Panavia F2.0 than for the Panavia group in which the mechanical modification of the zirconia surface was omitted (28.5 MPa vs. 8.9 MPa) [18].

In the current study, omitting the air-borne particle abrasion resulted in much lower SBS values. The same conclusion was drawn by other authors [2, 5, 16, 18, 22, 25, 27]. Moreover, they confirm not only the lower SBS values of zirconia to resin cements, but also the adhesive interface degradation due to the hydrolysis in case the alumina air-abrasion is omitted [22, 25]. The obtained results indicate that the optimal adhesive algorithm for resto-

rations made of zirconia-based ceramics would be the use of alumina air-borne particle abrasion prior to primer application. Although air-abrasion causes microstructural changes in the topmost layers of zirconia samples such as irregularities, grooves and plastic deformation, as well as tetragonal to monoclinic phase transformation, it has also many advantages [3, 9, 10, 15, 16, 19, 20, 30]. The positive outcomes of alumina air-abrasion are evident, as it increases bond strength of 3Y-TZP to resin cements, increases flexural strength of 3Y-TZP, improves marginal adaptation, prevents micro-leakage and removes contaminations [2, 5, 7, 13, 16, 18, 19, 22, 25, 27, 28].

The obtained results indicate that omitting air-borne alumina abrasion significantly decreases the SBS values of four resin cements to zirconia-based ceramics (mean 6.37 MPa). Treating the zirconia sample surface with air-abrasion resulted in the increase in shear bond strength to resin cements. The highest SBS values were yielded after using $50\ \mu\text{m}$ alumina particles (for Panavia F2.0, Multilink Automix, Duo-Link) – 11.59 MPa to 14.15 MPa. However, there was no statistical significance manifested in reference to the alumina particle

FIGURE 3. Representative SEM micrograph of the failure mode of zirconia surface/resin cement interface at $\times 35$ magnification of specimens treated with air-borne particle abrasion with alumina of different grades: **A)** untreated, **B)** 50 μm alumina particles, **C)** 110 μm alumina particles and **D)** 250 μm alumina particles, where Monobond Plus and Multilink Automix were used

grade used for air-borne particle abrasion. It is also confirmed by other research studies, although the alumina particle grade may be relevant for long-term durable bonding to zirconia [7, 22].

The results obtained in the shear bond strength test correlate with results of fractographic analysis of the fracture mode of the 3Y-TZP/resin cement interface. The higher the SBS values were, the more mixed, even cohesive failure modes were observed. Omitting the air-abrasion resulted in low SBS values, and for those specimens the failure mode was mainly adhesive. The failure mode was mixed for specimens that were treated with air-borne alumina abrasion. Moreover, in groups in which Duo-Link and Z-Prime Plus were used, the failure mode was even cohesive, as for those specimens the highest SBS values were obtained. Similar results were obtained by other researchers [5, 12, 13]. It is also worth mentioning that usually mixed and cohesive failure modes yielded after SBS testing occur in nearly 55% of the research groups. The adhesive failure modes usually are obtained when the SBS values are rather low [4]. The mixed and cohesive failure mode can be

considered as clinically favorable, as the adhesive forces exceeded the material cohesion [1, 4, 7].

Assuming all results obtained in this study, the most favorable cementation procedure that may be applied to zirconia-based ceramic restorations would be a two-stage procedure: first, air-borne particle abrasion with 50 μm alumina particles and then application of primer incorporating 10-MDP and carboxylic monomers, such as Z-Prime Plus primer.

CONCLUSIONS

The chemo-mechanical treatment of zirconia surface before adhesive cementation is crucial to obtain satisfactory values of shear bond strength to resin cements.

The highest shear bond strength of 3Y-TZP to resin cements is obtained after 50 μm alumina particle air-abrasion of the zirconia surface, although there is no statistical significance according to the alumina particle grade used for air-abrasion. Omitting alumina air-abrasion results in significantly decreased shear bond strength values.

The highest shear bond strength of 3Y-TZP to resin cements is obtained for Z-Prime Plus primer and Duo-Link resin cement.

CONFLICT OF INTEREST

The authors declare no potential conflicts of interest with respect to the research, authorship, and/or publication of this article.

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