ACP
AMERICAN COLLEGE OF PROSTHODONTISTS®

Durability of Resin Cement Bond to Aluminium Oxide and Zirconia Ceramics after Air Abrasion and Laser Treatment

Richard M. Foxton, BDS, PhD, MFDS RCS (Ed), FHEA,[1] Andrea N. Cavalcanti, DDS, MS, PhD,[2]
Masatoshi Nakajima, DDS, PhD, Peter Pilecki, BSc,[4] Martyn Sherriff, BSc, PhD,[4]
Luciana Melo, DDS, PhD,[1] & Timothy F. Watson, BDS, BSc, PhD, FDS, RCS[1,4]

[1] Department of Restorative Dentistry, King's College London Dental Institute at Guy's, King's and St. Thomas' Hospitals, King's College London, UK
[2] Department of Clinical Dentistry, School of Dentistry, UFBA, Salvador, Brazil
[3] Cariology and Operative Dentistry, Tokyo Medical and Dental University, Tokyo, Japan
[4] Department of Biomaterials, King's College London Dental Institute at Guy's, King's and St. Thomas' Hospitals, King's College London, UK

Keywords
Durability; resin cement; alumina; zirconia; adhesion; air abrasion; laser; bond strength.

Correspondence
Richard M. Foxton, Floor 25, Division of Conservative Dentistry, King's College London Dental Institute, Guy's Tower, Guy's Hospital, London Bridge, London, SE1-9RT, UK. E-mail: richard.foxton@kcl.ac.uk

Accepted April 17, 2010

doi: 10.1111/j.1532-849X.2010.00678.x

Abstract

Purpose: The erbium laser has been introduced for cutting enamel and dentin and may have an application in the surface modification of high-strength aluminum oxide and zirconia ceramics. The aim of this study was to evaluate the durability of the bond of conventional dual-cured resin cements to Procera Al_2O_3 and zirconium oxide ceramics after surface treatment with air abrasion and erbium laser.
Materials and Methods: One hundred twenty Al_2O_3 and 120 zirconia specimens measuring $3 \times 3 \times 0.7$ mm^3 were divided equally into three groups, and their surfaces treated as follows: either untreated (controls), air abraded with Al_2O_3 particles, or erbium-laser-treated at a power setting of 200 mJ. The surface of each specimen was then primed and bonded with one of two dual-cured resin cements (either SCP-100 Ceramic Primer and NAC-100 or Monobond S and Variolink II) using a 1-mm thick Tygon tube mold with a 0.75-mm internal bore diameter. After 24 hours and 6 months of water storage at 37°C, a microshear bond strength test was performed at a crosshead speed of 1 mm/min. Surface morphology was examined using a confocal microscope, and failure modes were observed using an optical microscope. The data were analyzed using the Kaplan-Meier nonparametric survival analysis.
Results: In the case of zirconia, air abrasion and Erbium:yttrium-aluminum-garnet (Er:YAG) laser treatment of the ceramic surface resulted in a significant reduction in the bond strengths of both resin cements after 6 months water storage; however, when the zirconia surface was left untreated, the SCP-100/NAC-100 group did not significantly reduce in bond strength. In the case of alumina, no treatment, air abrasion and Er:YAG laser treatment of the surface led to no significant reduction in the bond strengths of the three SCP-100/NAC-100 groups after 6 months water storage, whereas all three Monobond S/Variolink II groups showed a significant reduction.
Conclusion: Er:YAG laser treatment of the zirconia surface did not result in a durable resin cement/ceramic bond; however, a durable bond between a conventional dual-cured resin cement and Procera All Ceram and Procera All Zirkon was formed using a ceramic primer containing the phosphate monomer, MDP, without any additional surface treatment.

High-strength aluminum oxide and zirconia ceramics have been introduced to the dental profession as copings for the fabrication of metal-free, full-coverage crowns, and fixed dental prostheses.[1] If a method for reliably chemically bonding resin cement to alumina and zirconia could be achieved, then more tooth structure could be preserved, as less enamel and dentine would need to be removed to create retention and resistance form for the restoration. In addition, adhesive bonding of alumina and zirconia would enable teeth with short or reduced clinical crowns to be more reliably restored with such materials. Achieving a durable adhesive bond to these materials is difficult, because the absence of silicon dioxide makes them resistant to etching by hydrofluoric acid and not amenable to silanization.[2,3] A number of surface treatment techniques, such as sandblasting, tribochemical silica coating, use of a phosphate-monomer-containing resin cement, using a phosphate monomer-zirconate coupling agent, and more recently, a selective infiltration technique of the zirconia surface, have been reported.[4-8]

Durability studies of the bond between resin cements and high-strength Al_2O_3 and zirconia ceramics using long-term water storage and/or thermocycling are, however, limited. Hummel and Kern reported that resin cement could form a durable bond to Procera alumina ceramic using a combination of sandblasting and a 10-methacryloyloxydecyl dihydrogen phosphate- (MDP) containing primer or sandblasting and the inclusion of MDP in the resin cement.[6] Wegner and Kern reported that a durable bond to zirconia ceramic could be achieved using a combination of air abrasion and a phosphate-monomer-containing resin cement;[9] however, Blatz et al highlighted the fact that alumina and zirconia ceramics from different manufacturers are often used in in vitro studies along with different resin cements, and suggested that caution should be applied when comparing such studies and extrapolating the results to the clinical situation.[10]

Al_2O_3 particles with sizes ranging from 25 to 250 μm are commonly used for air abrading the ceramic surface. These particles might or might not be silica coated.[11] The abrasive process removes loose contaminated layers, increases the area available for bonding, and improves the wettability of luting materials.[12,13] Nevertheless, flaws created by air abrasion may function as crack initiators in Y-TZP materials, compromising their mechanical properties and long-term performance.[14] Contrasting results observed with air-abraded Y-TZP ceramics indicate that the effects of air abrasion and other methods of surface modification, such as laser irradiation, should be further investigated.

The Erbium:yttrium-aluminium-garnet (Er:YAG) laser has been proposed for different clinical dentistry applications, including carious dentin removal, cavity preparation, and as a surface treatment method for indirect restorations made of lithium disilicate and composites.[15-19] In tooth substrates, the Er:YAG laser produces microexplosions during hard tissue ablation, resulting in macroscopic and microscopic irregularities that may constitute a surface for adhesion.[15] Although plenty of information regarding the effects of Er:YAG irradiation on dentin and enamel structures exists, little is known about the use of this laser as a surface treatment for bonding resin cement to high-strength dental ceramics.

The aim of this experiment was to investigate the durability of the bond between two conventional dual-cured resin cements and high-strength Al_2O_3 and zirconia ceramics after no surface treatment and treatment with either air abrasion or Er:YAG laser. The null hypothesis was that the type of surface treatment would have no statistically significant effect on the durability of the resin/ceramic bond, at a significance of $\alpha = 0.05$.

Materials and methods

The composition of the resin cements and ceramic primers used in the present experiment are described in Table 1. Blocks of densely sintered aluminum oxide (Procera AllCeram, Nobel Biocare, Göteborg, Sweden) and densely sintered zirconium oxide (Procera AllZirkon, Nobel Biocare) ceramic measuring $10 \times 10 \times 5$ mm^3, were cut into 240, $3 \times 3 \times 0.7$ mm^3 specimens using a diamond wafering blade (high concentration, XL 12205, Benetec Limited, London, UK), mounted in an Isomet low-speed cutting saw (Buehler, Coventry, UK), and divided into two groups: 120 alumina and 120 zirconia specimens. All specimens were then ultrasonically cleaned with 96% isopropanol for 3 minutes. Each group was randomly subdivided into three groups of 40 specimens for each surface treatment: none (control), air abrasion, or Er:YAG laser (Fig 1).

Surface treatment

For the control groups, no additional surface treatment was performed after cleaning with isopropanol. In the air-abraded and lased groups, the superficial area to be further treated (1.76 mm^2) was outlined with adhesive tape.

Air abrasion was performed with 53-μm Al_2O_3 particles (Aquacut, Medivance Instruments Ltd., London, UK) at a 2.5 bar pressure for 15 seconds at a distance of 10 mm. After air

Table 1 Chemical composition of the resin cements and ceramic primers

Material	Manufacturer	Lot no.	Principal ingredients
NAC-100 (Universal, now marketed as Clearfil Esthetic Cement)	Kuraray Medical Incorporated, Tokyo, Japan	050616	Paste A: Bis-GMA, TEGMA, methacrylate monomers, silanated glass filler, colloidal silica. Paste B: Bis-GMA, TEGMA, methacrylate monomers, silanated glass filler, colloidal silica, Benzoyl peroxide, dl-camphorquinone, pigments
Variolink II (transparent, base, and catalyst)	Ivoclar Vivadent AG, Schaan, Liechtenstein	H22495 G18209	Bis-GMA, UDMA, TEGMA, barium glass, ytterbium trifluoride, Ba-F-fluorosilicate glass, catalysts, stabilizers, and pigments
SCP-100 (now marketed as Clearfil Ceramic Primer)	Kuraray Medical Incorporated	15 K	MPTS, MDP, Ethanol
Monobond-S	Ivoclar Vivadent AG	H22376	MPTS, Water/Ethanol, Acetic acid

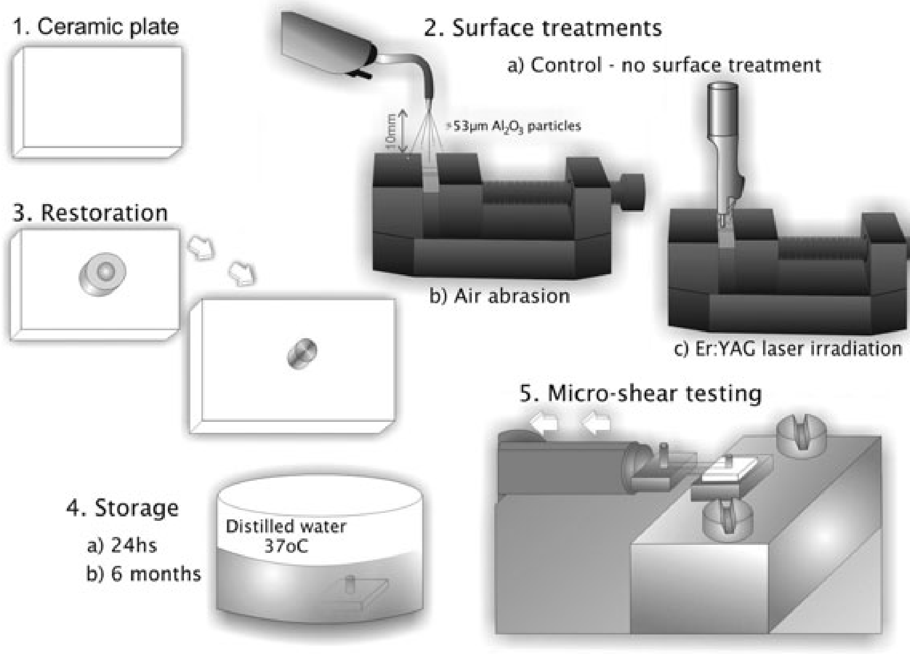

Figure 1 Schematic diagram of the experiment set-up.

abrasion, the adhesive tape was removed, and the plates were ultrasonically cleaned with 96% isopropanol for 3 minutes.

Prior to laser irradiation, surfaces were coated with graphite to increase the absorption of energy.[20] The laser equipment used was an Er:YAG laser (OPUS 20 Er:YAG/CO_2 Dental Laser Surgical System, Sharplan Medical Systems, Yokneam, Israel) emitting a 2.94-μm wavelength. A 1000-μm-diameter straight-type sapphire tip, which was held by hand, was used perpendicular to the surface in contact mode. The surfaces were lased for 5 seconds using a fine water spray during operation. The water spray was supplied by the laser equipment, the pulse repetition rate was set at 10 Hz, and the energy intensity was 200 mJ. After the irradiation, the adhesive tape was removed, and surfaces were ultrasonically cleaned in 96% isopropanol for 3 minutes.

Microshear bond strength test

For each surface treatment (controls, air-abrasion, laser) 20 specimens were treated with an experimental ceramic primer, SCP-100, and 20 specimens with the ceramic primer, Monobond-S, according to the manufacturers' instructions. A 1-mm thick slice of Tygon tubing (TYG-030, Small Parts Inc., Miami Lakes, FL) with a 0.75-mm internal bore diameter was then filled with one of two resin cements (an experimental resin cement, NAC-100, or Variolink II) and bonded to the ceramic surface.[21] Light-activated polymerization was performed for 20 seconds using a halogen light-curing unit (Optilux, Demetron Research Corporation, Danbury, CT), whose power density was checked prior to use.

The prepared specimens were randomly allocated according to whether storage was to be for 24 hours or 6 months in water at 37°C. Each group consisted of 10 specimens. To eliminate the effect of Tygon tube removal on the adhesion of the composite-ceramic specimens, the surrounding Tygon tubing was carefully removed from all the specimens after 24 hours using a scalpel.

After 24 hours or 6 months water storage, each ceramic plate was fixed in a microshear device adapted in a universal testing machine (SMAC LAL95, SMAC Europe, Horsham, Sussex, UK) with cyanoacrylate adhesive (SuperGlue, Loctite, Henkel Loctite, Hertfordshire, UK). A thin wire (0.2 mm diameter) was looped around the resin cylinder, making contact with half its circumference at the resin/ceramic interface. A shear force was applied at a crosshead speed of 1 mm/min until debonding. After debonding, the fractured surfaces were evaluated with an optical microscope (30 × magnification) to classify the failure modes into one of the following categories: (A) adhesive failure at the interface between the ceramic and resin; (B) cohesive failure within resin; and (C) cohesive failure in ceramic.

Surface morphology examination

Three additional alumina and three zirconia plates were examined using confocal microscopy to evaluate the ceramic topography after the surface treatments. For each surface treatment group, two ceramic plates were treated and cleaned as described previously, followed by gold-sputter coating (E5100, Polaron Equipment Ltd., Hertfordshire, UK) to create opaque surfaces for topographic recording. Each specimen was observed with a tandem scanning confocal microscope (TSM) (Noran Instruments, Middleton, WI), using an x100/1.40 NA oil immersion lens with a 546-nm illumination filter to reduce chromatic aberration. Using an automatic stage controller (Märzhäuser,

Table 2 Mean microshear bond strengths with standard deviations (MPa) and failure modes

								Locus		
Code	Cement	Ceramic	Storage	Treatment	n	Mean	SD	L1	L2	L3
1	Variolink II	Alumina	1d	None	0	0	0	0	10	0
2	Variolink II	Alumina	1d	Air abrasion	4	17.63	4.29	0	8	2
3	Variolink II	Alumina	1d	Laser	2	15.15	0.21	0	10	0
4	Variolink II	Alumina	6m	None	2	11.00	0.00	0	10	0
5	Variolink II	Alumina	6m	Air abrasion	7	9.01	1.18	0	10	0
6	Variolink II	Alumina	6m	Laser	4	10.35	2.32	0	10	0
7	Variolink II	Zirconia	1d	None	5	18.94	2.95	0	7	3
8	Variolink II	Zirconia	1d	Air abrasion	5	20.98	3.69	0	6	4
9	Variolink II	Zirconia	1d	Laser	2	13.95	0.92	0	10	0
10	Variolink II	Zirconia	6m	None	2	14.65	0.49	0	10	0
11	Variolink II	Zirconia	6m	Air abrasion	3	8.97	2.76	0	10	0
12	Variolink II	Zirconia	6m	Laser	4	8.3	1.15	0	10	0
13	NAC 100	Alumina	1d	None	2	17.3	0.99	0	10	0
14	NAC 100	Alumina	1d	Air abrasion	8	17.53	1.10	0	5	5
15	NAC 100	Alumina	1d	Laser	7	16.05	1.33	0	5	5
16	NAC 100	Alumina	6m	None	5	12.72	3.92	0	10	0
17	NAC 100	Alumina	6m	Air abrasion	8	25.68	7.64	0	6	4
18	NAC 100	Alumina	6m	Laser	3	16.73	8.44	0	8	2
19	NAC 100	Zirconia	1d	None	8	19.08	2.14	0	6	4
20	NAC 100	Zirconia	1d	Air abrasion	8	19.55	2.02	0	5	5
21	NAC 100	Zirconia	1d	Laser	7	16.27	4.14	0	6	4
22	NAC 100	Zirconia	6m	None	5	19.84	3.82	0	7	3
23	NAC 100	Zirconia	6m	Air abrasion	9	14.79	2.13	0	6	4
24	NAC 100	Zirconia	6m	Laser	4	10.93	3.08	0	8	2

Legend: Storage 1d = 1 day; 6m = 6 months; n = Number of specimens tested (maximum = 10); Mean = Mean bond strength/MPa; SD = Standard deviation/MPa; Locus = Locus of failure of pretest and tested specimens: L1 = Cohesive in ceramic; L2 = Interfacial adhesive; L3 = Cohesive in cement.

Wetzlar-Steindorf, Germany), a 4912 monochrome CCD camera (Cohu Inc., San Diego, CA), and image-capturing software (AQM 6, Andor Technology, Belfast, UK), the surfaces were optically profiled by sequentially capturing surface images from the highest to the lowest planes of focus, with a step interval of 0.2 μm. Each captured image stack was processed (Lucida Analyse, Andor Technology) to obtain a single image displaying the brightest points in each optical section, thus producing a view of the specimen's topography.

Statistical analysis

The bond strength data were analyzed using Stata version 9 (StataCorp LP, College Station, TX) with significance predetermined at $\alpha = 0.05$. The Kaplan-Meier nonparametric analysis was used to estimate the survival function, and the log-rank test was used to compare the survival distributions.

Results

The maximum number of specimens available for testing was 240, 10 per group; however, 127 failed adhesively at the ceramic interface during removal of the Tygon tubing prior to testing. These pretest failures were included in the survival analysis. The means and standard deviations of the obtained microshear bond strengths and the number of survival specimens are summarized in Table 2 and Figure 2. The majority of the specimens failed adhesively at the resin/ceramic interface after both 1 day and 6 months water storage (Table 2). The results of the Kaplan-Meier nonparametric analysis and the log-rank test are shown in Tables 3 and 4, and Figures 3-6.

In the case of Variolink II, for both alumina and zirconia there were significant differences between the three surface treatments (control, air abrasion, laser) after both 1 day and 6 months water storage (Figs 3 and 4). In the case of Variolink II bonded to alumina, there was a statistically significant reduction in bond strength following surface treatment with either air abrasion or laser after 6 months water storage. For Variolink II bonded to zirconia, there was a significant reduction in bond strength following no surface treatment, and surface treatment with air abrasion, and laser after 6 months water storage.

In the case of NAC-100 bonded to alumina, there appeared to be no significant difference between the three surface treatments (control, air abrasion, laser) after both 1 day and 6 months water storage (Fig 5). In the case of NAC-100 bonded to alumina, there was no significant reduction in bond strength between the control groups and surface treatment with air abrasion and laser after 6 months water storage.

Regarding NAC-100 bonded to zirconia, there was no significant difference between the three surface treatments (control, air abrasion, laser) after 1 day of water storage; however, after 6 months water storage, there was a significant reduction

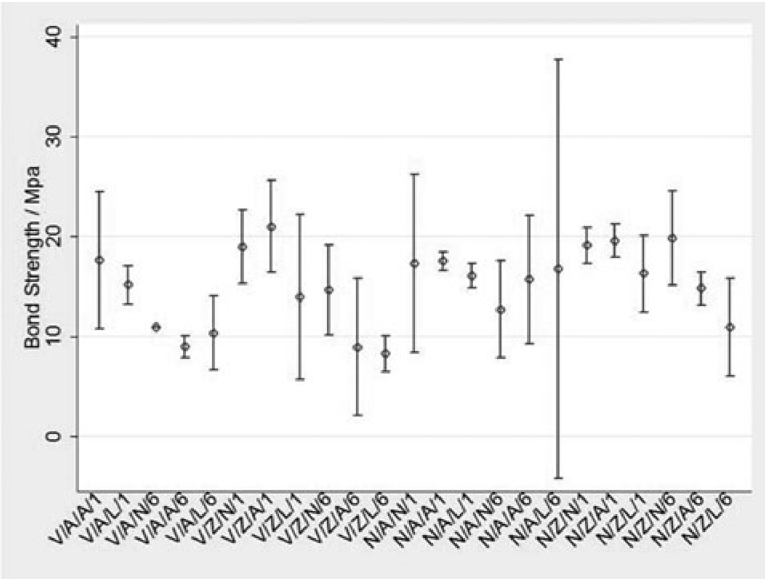

Figure 2 Bond strengths after 1 day and 6 months of water storage: A = alumina; V = zirconia; N = no; A = air abrasion; L = laser; 1 = 1 day; 6 = 6 months.

in bond strength after surface treatment with air abrasion and laser. Whereas for the control group, bond strengths did not significantly reduce (Fig 6).

With regards to the confocal observations of the treated surfaces of the alumina and zirconia specimens, air abrasion did not appear to significantly alter the appearance of either alumina or zirconia (Figs 7 to 10); however, a clear topographic effect of laser treatment was demonstrated, manifesting as a damaged, roughened appearance, particularly including cracking in zirconia. Dark, confluent areas were apparent in the scans (Figs 11 and 12).

Discussion

The present experiment examined whether Er:YAG laser treatment of the surfaces of densely sintered alumina and zirconia ceramic would result in a durable dual-cure resin ceramic bond. This was compared with air abrasion using Al_2O_3 particles and no additional surface treatment after applying the manufacturers' recommended ceramic primers.

In the case of alumina after surface treatment with air abrasion, previous research has shown that a durable bond can be obtained between a conventional resin cement (Variolink II) and air-abraded alumina if a primer containing a phosphate monomer or silane coupling agent is used.[10] In this study, the Variolink II group significantly reduced in bond strength after 6 months, whereas the NAC-100 group did not. The confocal micrographs showed that air abrasion had no obvious discernable effect on the alumina surface when compared to the untreated surface (Figs 7 and 8). Neither resin cement contains a phosphate monomer, but the primer SCP-100 contains MDP. Therefore, the results of the present study partially agree with previous research in the case of the combination SCP-100 and NAC-100 but disagree with regards to the combination of Monobond S and Variolink II providing a durable bond to

Table 3 Median survival bond strength

System	Substrate	Treatment	1 day N	1 day BS_{50}	6 months N	6 months BS_{50}
Variolink	Alumina	None	0		2	11
		Air	4	14.3	7	9
		Laser	2	15	4	10
	Zirconia	None	5	20.6	2	14.3
		Air	5	19	3	8.3
		Laser	2	13.3	4	7.6
NAC-100	Alumina	None	2	16.6	5	14.2
		Air	8	17.3	8	17.2
		Laser	7	15.6	3	21.2
	Zirconia	None	8	18.6	5	21
		Air	8	19.6	9	15.3
		Laser	7	15	4	9.2

N = number of specimens tested; BS_{50} = Median bond strength/MPa.

Table 4 Log-rank test for the effect of storage on survivor function

System	Substrate	Treatment	$Pr > \chi^2$
Variolink	Alumina	None	
		Air	0.0039
		Laser	0.0494
	Zirconia	None	0.0082
		Air	0.0042
		Laser	0.0439
NAC-100	Alumina	None	0.1526
		Air	0.5331
		Laser	0.1331
	Zirconia	None	0.3800
		Air	0.0002
		Laser	0.0132

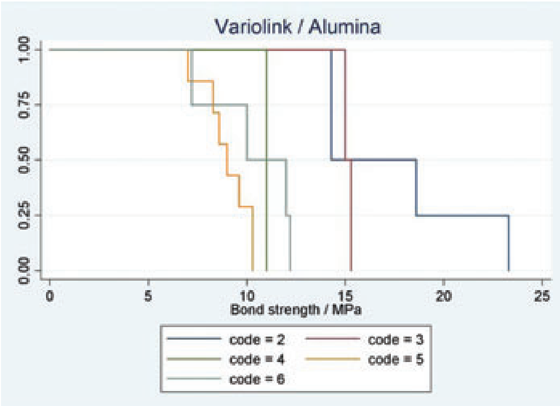

Figure 3 Kaplan-Meier survival analysis of Variolink II bonded to alumina.

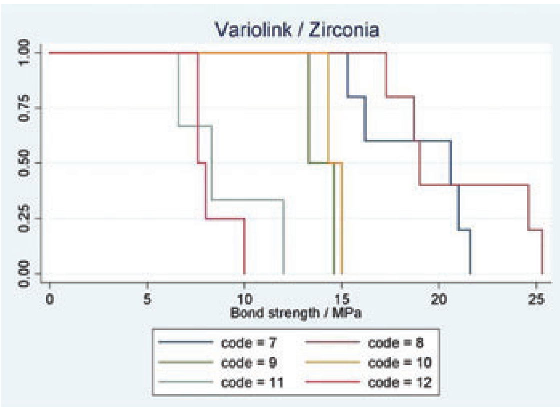

Figure 4 Kaplan-Meier survival analysis of Variolink II bonded to zirconia.

air-abraded alumina. It is therefore indicated that if the resin cement does not contain a phosphate monomer such as MDP, a primer containing such a monomer be used.

With regards to the durability of the resin cement/zirconia bond after air abrasion of the surface with 53-μm Al_2O_3 particles, it has been demonstrated that when conventional resin cement was bonded to zirconia whose surface had been air abraded and primed using a ceramic primer containing the phosphate monomer MDP, bond strengths were not significantly reduced after long-term water storage;[10] however, when

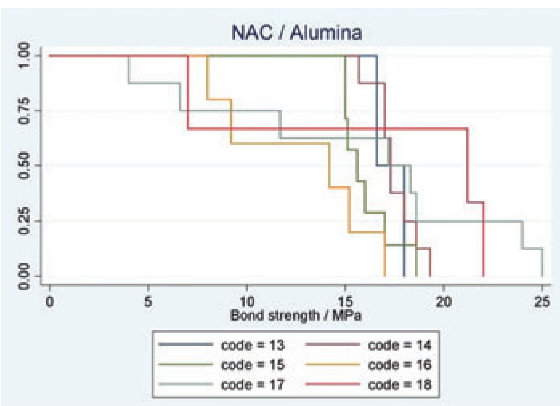

Figure 5 Kaplan-Meier survival analysis of NAC-100 bonded to alumina.

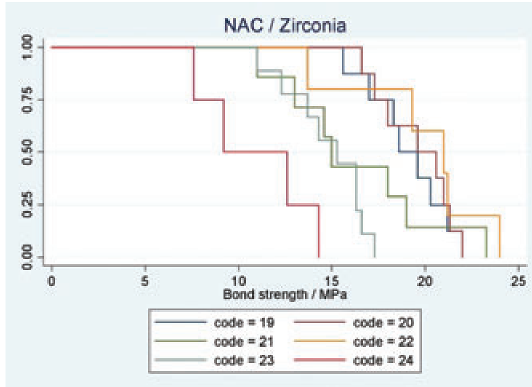

Figure 6 Kaplan-Meier survival analysis of NAC-100 bonded to zirconia.

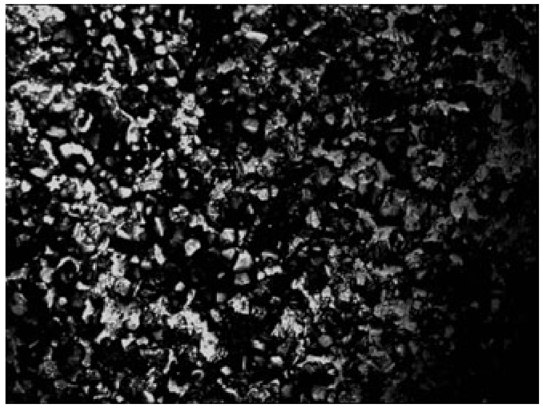

Figure 7 Representative topographic confocal scanning image of the control alumina surface. TSM × 100/1.40 NA oil immersion objective. Fieldwidth 100 μm.

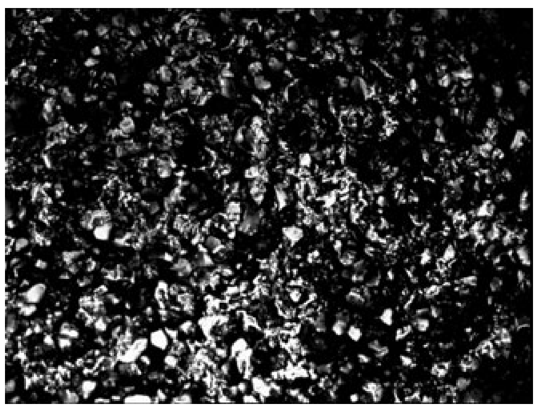

Figure 8 Representative topographic confocal scanning image of the alumina surface after air abrasion. The surface is not notably altered from the control state. TSM × 100/1.40 NA oil immersion objective. Fieldwidth 100 μm.

Figure 9 Representative topographic confocal scanning image of the control zirconia surface. The surface has clear sectioning marks, which were created during specimen preparation. TSM $\times$ 100/1.40 NA oil immersion objective. Fieldwidth 100 μm.

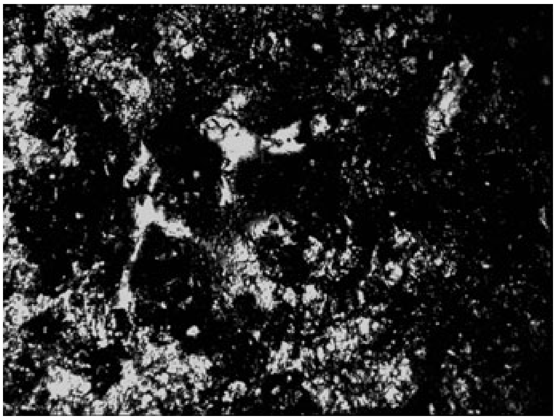

Figure 11 Representative topographic confocal scanning image of the alumina surface after Erbium laser treatment. The surface morphology is altered, showing a more complex topography involving smoothened surfaces. TSM $\times$ 100/1.40 NA oil immersion objective. Fieldwidth 100 μm.

the zirconia surface was air abraded with 50-μm particles and bonded with a resin cement that did not contain a phosphate monomer, a stable bond was not formed.[22] Both dual-cure resin cements used in the present study did not contain a phosphate monomer, but the ceramic primer SCP-100 contained MDP; however, both the Variolink II/Zirconia and NAC-100/Zirconia groups significantly reduced in bond strength after 6 months water storage. In addition, the confocal micrographs showed that air abrading the zirconia surfaces had no significantly noticeable effect when compared to the control specimens (Figs 9 and 10). The results of the present experiment disagree with the findings of Blatz but agree with those of Wolfart, which in both cases used alumina particles with a similar size to those in the present experiment.[10,22] It is therefore indicated that when a resin cement that does not contain a phosphate is used for bonding to zirconia, the formation of a durable bond may depend on the ceramic primer used.

To date, there has been no research published on the durability of resin-alumina/resin-zirconia bonds after surface treatment with an Er:YAG laser. Concerning the alumina groups, after 6 months water storage, there was no significant difference in bond strengths between the control and Er:YAG groups when bonded with NAC-100, and in the case of Variolink II, the bond strengths were similar. When the confocal micrographs of the surfaces of the untreated and lased alumina specimens are compared, the alumina crystal boundaries have disappeared, and many more confluent areas are visible (Figs 7 and 11). These results indicate that adhesion was not improved by surface treatment with Er:YAG laser when compared to no surface treatment prior to application of the primer.

In the case of Procera All Zirkon, both groups exhibited a significant reduction in bond strength after 6 months water storage. The effect of Er:YAG laser has been investigated on

Figure 10 Representative topographic confocal scanning image of the zirconia surface after air abrasion. There is little alteration of the surface texture, but the surface is more randomly marked than the control. TSM $\times$ 100/1.40 NA oil immersion objective. Fieldwidth 100 μm.

Figure 12 Representative topographic confocal scanning image of the zirconia surface after Erbium laser treatment. The surface morphology is very different after laser treatment, demonstrating surface penetration and smoother areas with extensive cracking. TSM $\times$ 100/1.40 NA oil immersion objective. Fieldwidth 100 μm

lithia-based all-ceramic material.[16] The authors examined the effect of Er:YAG laser at three laser power settings (300, 600, 900 mJ) on the bond strength of Variolink II to lithia-based all-ceramic and found that a power setting of 300 mJ created the optimum etching pattern, irregular surface, and bond strength.[16] In this study, an energy intensity of 200 mJ was selected following our previous study on the effect different laser intensities have on surface morphology and roughness of two zirconia ceramics.[20] In our previous study, higher laser power settings of 400 mJ and 600 mJ were found to cause melting, loss of surface material, and deep cracks. A power setting of 200 mJ was found to cause less melting, solidification of the surface, and cracking.[20] These observations were also evident in the present experiment. A damaged, roughened appearance, particularly including cracking in zirconia, was evident following laser treatment at a power setting of 200 mJ. The dark, confluent areas probably indicated melting and subsequent cooling after applying the laser (Figs 11 and 12). The resultant increased surface area offers the potential for improved bonding, but this was not supported by the bond strength results. The increased overall surface area may have been compromised by the reformed areas, which appeared smoother than the control surfaces. The results of the present experiment therefore appear to indicate that Er:YAG treatment of zirconia ceramic may not be an appropriate surface treatment for obtaining a durable bond to resin cement.

A pilot study was initially performed in which the resin cement was sandwiched between either two alumina or two zirconia blocks. The bonded specimens were then sliced into slabs and then beams for the microtensile bond strength test. All the beams debonded prior to testing. This was independently validated by another experienced researcher. It was therefore considered that the microtensile test was not appropriate for this experiment, because the hardness of alumina and zirconia prevented fast movement of the cutting blade through the resin/ceramic interface, resulting in a fatiguing effect on the beams, which resulted in them debonding during slicing. Therefore the microshear test was selected for the present study.[21] This still allowed for bonded specimens with a small cross-sectional area to be obtained, allowing for a greater potential "aging effect" when stored in water; however, a high number of specimens failed despite careful removal of the Tygon tubing mold. These failures were all adhesive, indicating that the small cross-sectional area of the specimens may have rendered the resin/ceramic bond susceptible to failure if its bond strength was low, although the resin cement itself was strong enough to withstand specimen preparation.

Variolink II and NAC-100 are conventional dual-cured resin cements with no acidic phosphate monomer; little research has been conducted on the adhesion of these types of cements in conjunction with phosphate-monomer-containing ceramic primers to surface-modified alumina and zirconia. Previous research has investigated the effect of 50-μm Al_2O_3 particles on the strength of Y-TZP and alumina ceramic layers when loaded.[23] The strengths of the sandblasted specimens showed significant reductions in both dynamic and cyclic tests when compared with the polished specimens, and the authors concluded that surface abrasion treatments can be an important degrading factor in the long-term performance of all-ceramic crowns.[23] In this experiment, the control alumina and zirconia specimens did not receive any surface treatment with either Er:YAG laser or air abrasion, and there was no significant reduction in bond strength for the NAC groups. In the case of Procera alumina ceramic, previous research reported that when Variolink II was bonded to alumina using a metal primer containing MDP (Alloy Primer) without any additional surface treatment, initial bond strengths were low, and a stable bond was not formed.[6] The results of this study for NAC-100 are contrary to those findings, as NAC-100 was able to form a stable bond to alumina and zirconia with a ceramic primer containing MDP without any additional surface treatment. Unfortunately, there has been very little research published on this subject, and therefore further research using different phosphate-monomer-containing primers and resin cements is needed.

To date, no research has been published on the durability of the bond between non-phosphate-monomer-containing, dual-cured resin cements, and zirconia using a primer containing MDP. Previous research has shown good initial bonding between nonphospahte monomer, dual-cured resin cements, and zirconia when a phosphate-monomer-containing ceramic primer was used.[7,24,25] Moreover, our previous study examined the effect of different surface treatments on the surface roughness of Procera All Zirkon ceramic, and it was found that air abrasion did not create a significantly rougher surface than the control group.[20] The results of this experiment therefore indicate that if a primer containing a phosphate monomer is used on an alumina or zirconia ceramic surface, additional treatment with air abrasion may not be necessary. In addition, there appears to be no apparent clinical advantage in carrying out any physical surface treatment of alumina core ceramics, and this may weaken long-term resin bond strength for zirconia core ceramics.

Conclusions

1. Er:YAG laser treatment of the zirconia surface did not result in a durable resin cement/ceramic bond. In the case of alumina, surface treatment with Er:YAG laser did not result in an improvement in bond strengths compared with the air-abraded and untreated specimens.
2. A durable bond to air-abraded alumina was formed when conventional dual-cured resin cement was used in conjunction with a ceramic primer containing the phosphate monomer, MDP; however, this was not the case for air-abraded zirconia.
3. When the alumina and zirconia specimens were left untreated, a durable bond was formed to both materials when the surfaces were treated with a ceramic primer containing the phosphate monomer MDP and bonded with conventional dual-cured resin cement.

References

1. Sadan A, Blatz MB, Lang B: Clinical considerations for densely sintered alumina and zirconia restorations: part 2. Int J Perio Rest Dent 2005;25:343-349
2. Friederich R, Kern M: Resin bond strength to densely sintered alumina ceramic. Int J Prosthodont 2002;15:333-338

3. Dérand P, Dérand T: Bond strength of luting cements to zirconium oxide ceramics. Int J Prosthodont 2000;13: 131-135
4. Kern M, Thompson VP: Sandblasting and silica coating of a glass-infiltrated alumina ceramic: volume loss, morphology, and changes in the surface composition. J Prosthet Dent 1994;71:453-461
5. Kern M, Wegner SM: Bonding to zirconia ceramic: adhesion methods and their durability. Dent Mater 1998;14:64-71
6. Hummel M, Kern M: Durability of the resin bond strength of the alumina ceramic Procera. Dent Mater 2004;20:498-508
7. Yoshida K, Tsuo Y, Atsuta M: Bonding of dual-cured resin cement to zirconia ceramic using phosphate acid ester monomer and zirconate coupler. J Biomed Mater Res B Appl Biomater 2006;77:28-33
8. Aboushelib MN, Kleverlaan CJ, Feilzer AJ: Selective infiltration-etching technique for a strong and durable bond of resin cements to zirconia-based materials. J Prosthet Dent 2007;98:379-388
9. Wegner SM, Kern M: Long-term resin bond strength to zirconia ceramic. J Adhes Dent 2000;2:139-147
10. Blatz MB, Sadan A, Martin J, et al: In vitro evaluation of shear bond strengths of resin to densely-sintered high purity zirconium-oxide ceramic after long-term storage and thermal cycling. J Prosthet Dent 2004;91:356-362
11. Curtis AR, Wright AJ, Fleming GJ: The influence of surface modification techniques on the performance of a Y-TZP dental ceramic. J Dent 2006;34:195-206
12. Amaral R, Ozcan M, Bottino MA, et al: Microtensile bond strength of a resin cement to glass infiltrated zirconia-reinforced ceramic: the effect of surface conditioning. Dent Mater 2006;22:283-290
13. Kumbuloglu O, Lassila LV, User A, et al: Bonding of resin composite luting cements to zirconium oxide by two air-particle abrasion methods. Oper Dent 2006;31:248-255
14. Zhang Y, Lawn BR, Rekow ED, et al: Effect of sandblasting on the long-term performance of dental ceramics. J Biomed Mater Res B Appl Biomater 2004;71:381-386
15. Aranha AC, Turbino ML, Powell GL, et al: Assessing microleakage of class V resin composite restorations after Er:YAG laser and bur preparation. Laser Surg Med 2005;37:172-177
16. Gokce B, Ozpinar B, Dundar M, et al: Bond strengths of all-ceramics: acid vs laser etching. Oper Dent 2007;32:173-178
17. Burnett LH, Jr., Shinkai RS, Eduardo Cde P: Tensile bond strength of a one-bottle adhesive system to indirect composites treated with Er:YAG laser, air abrasion, or fluoric acid. Photomed Laser Surg 2004;22:351-356
18. Bader C, Krejci I: Indications and limitations of Er:YAG laser applications in dentistry. Amer J Dent 2006;19:178-186
19. van As G: Erbium lasers in dentistry. Dent Clin North Am 2004;48:1017-1059
20. Cavalcanti A, Pilecki P, Foxton RM, et al: Evaluation of the surface roughness and morphologic features of Y-TZP ceramics after different surface treatments. Photomed Laser Surg 2009;27:1-7
21. Shimada Y, Yamaguchi S, Tagami J: Micro-shear bond strength of dual-cure resin cement to glass ceramics. Dent Mater 2002;18:380-388
22. Wolfart M, Lehmann P, Wolfart S, et al: Durability of the resin bond strength to zirconia ceramic after using different surface conditioning methods. Dent Mater 2007;23:45-50
23. Zhang Y, Lawn BR, Rekow ED, et al: Effect of sandblasting on the long-term performance of dental ceramics. J Biomed Mater Res Part B: Appl Biomater 2004;71:381-386
24. Cavalcanti A, Foxton RM, Watson TF, et al: Y-TZP Ceramics: key concepts for clinical application. Oper Dent 2009;34:344-351
25. Cavalcanti A, Foxton RM, Watson TF, et al: Bond strength of resin cements to a zirconia ceramic with different surface treatments. Oper Dent 2009;34:280-287