Dental Materials Journal 2021; 40(1): 245–252

Effect of potassium hydrogen difluoride in zirconia-to-resin bonding

Jenni HJERPPE[1,2], Leila PEREA-LOWERY[3], Lippo V.J. LASSILA[3] and Pekka K. VALLITTU[3,4]

[1] *Department of Prosthetic Dentistry and Stomatognathic Physiology, Institute of Dentistry University of Turku, Lemminkäisenkatu 2, 20520 Turku, Finland*
[2] *Departments of Oral and Maxillofacial Diseases, Helsinki University Hospital (HUH), P.O. Box 263, FIN-00029 HUS, Finland*
[3] *Department of Biomaterials Science and Turku Clinical Biomaterials Centre —TCBC, Institute of Dentistry, University of Turku, Lemminkäisenkatu 2, 20520 Turku, Finland*
[4] *City of Turku, Welfare Division, Lemminkäisenkatu 2, 20520 Turku, Finland*
Corresponding author, Jenni HJERPPE; E-mail: jenni.hjerppe@utu.fi

The objective of this study was to compare potassium hydrogen difluoride (KHF_2) etching for zirconia with commonly used surface roughening and chemical bonding methods (silane, MDP-monomer primer) for resin-based luting cement bonding to zirconia. Zirconia specimens were divided into six groups (n=10) according to surface treatment and bonding procedures, with and without thermocycling (6,000 cycles, 5–55ºC): 1) air-borne particle abrasion with alumina+MDP-monomer (ABP), 2) air-borne particle abrasion with silica-coated trialuminium trioxide+silane (ABPR-S) and 3) KHF_2 etching+silane (ETC). Surface roughness and bond strength (SBS-test) for dry and thermocycled specimens were measured. SBS did not vary statistically between the dry groups, but thermocycling decreased the bond strengths of all the tested methods (p<0.05). After thermocycling, ABP had statistically significantly lower bond strength values compared to ABPR-S and ETC (p<0.05). Etching method with KHF_2 did not provide better bonding capacity to previously introduced and commonly adopted bonding methods.

Keywords: Zirconia, Bonding, Air-borne particle abrasion, Etching, Shear bond strength, Potassium hydrogen difluoride

INTRODUCTION

Partially yttrium oxide stabilized zirconium dioxide, zirconia, is used as a framework material for tooth- and implant-supported single crowns and fixed dental prostheses with a clinical history of over 10 years[1-3]. High quasi-static flexural strength is one of the properties which justify the use of zirconia as a dental material but some concerns have arisen with regard to its low fracture toughness compared to metal, and difficulty in obtaining durable and hydrolytically stable interfacial adhesion to resin composite luting cements. In a five-year follow-up study, zirconia crowns demonstrated more loss of retention than other all-ceramic or metal-ceramic crowns[4]. This could have been due to the fabrication methods used in early stage CAD/CAM systems, which might have lead to inaccuracies in the fitting of these restorations. Although, due to limitations in the chemical and micromechanical adhesion of resin composite luting cements to zirconia, the clinical success of using zirconia in resin bonded restorations has been mixed and more technique sensitive[5-7].

Different methods have been introduced for improving the interfacial adhesion of resin-based materials to zirconia, either by chemical or micromechanical means[8-12]. Silane coupling agents are often used for improving the surface wettability of ceramic surfaces by using resin composite luting cements and to provide covalent bonding of the cement to zirconia *via* spontaneously formed hydroxyl group coverage on the surface of silica based ceramics[13]. Silane coupling agents have limited influence with zirconia due to lack of a sufficient number of hydroxyl groups on the surface of zirconia. This leads to limited hydrolytic stability of the interfacial adhesion and reduction of bond strength between ziroconia and resin composites[14]. In laboratory studies, the bond strength (shear bond strength test; SBS) of zirconia has varied between 5.8–38.7 MPa depending on the testing method used[8,12,15,16]. At the moment the "gold standard" and most widely used method for the successful bonding of zirconia involves the air-borne particle abrasion of zirconia surfaces with aluminum oxide particles (particle size of 50 μm). This process increases the surface microroughness and allows micromechanical interlocking of the cement. Additionally, chemical means to adhere zirconia to resin composite cements have become of interest and one commonly used compound for promoting resin adhesion to zirconia is 10-methacryloyloxydecyl dihydrogen phosphate (MDP) containing primer[9]. Although MDP has successfully been used as an adhesion promoting agent based on covalent bonding of MDP to zirconia surfaces, there is a concern of hydrolytic degradation of the adhesive interface by the covalent bond or from the ester group of the MDP[17]. The limitations of this kind of bonding, have lead to the development of systems which provide a surface microroughness profile similar to that of etched glass ceramics, *i.e.* having high valley depth (R_v). A recently introduced etching system for zirconia by Ruyter *et al.* showed considerably enhanced bonding characteristics for resin luting cements[11]. In the study by Ruyter and colleagues, the zirconia surface was

Color figures can be viewed in the online issue, which is available at J-STAGE.
Received Nov 19, 2019: Accepted Apr 6, 2020
doi:10.4012/dmj.2019-389 JOI JST.JSTAGE/dmj/2019-389

etched at elevated temperature with either potassium hydrogen difluoride (KHF_2) or ammonium hydrogen difluoride (NH_4HF_2). The test of the interfacial bond strength was based on pure shear stress, which differs considerably from the commonly used SBS test where there is also a tensile stress component at the interface during the loading event. Therefore, the study by Ruyter *et al.* simulated only the clinical situation where zirconia crown was cemented on an implant abutment. The results cannot be used to justify the use of the bonding system, *e.g.* in a situation with resin bonded fixed dental prostheses where there will be considerable tensile stress components at the interface. In another recent study the shear bond strength test was used to evaluate the etching method of Ruyter and colleagues', but the results were not compared to the "gold standard" method of using MDP-monomer containing primer[18].

Thus, the objective of this study was to compare the shear bond strength (SBS) of zirconia specimens treated with the novel Ruyter etching system with the commonly used air-borne particle abrasion surface roughening system and chemical adhesion methods (silane or MDP-monomer containing primer) for resin-based luting cements. The null hypothesis was that there is no difference in interfacial adhesion strength between zirconia and resin luting cement between the different surface roughening methods when the testing is made using the SBS test.

MATERIALS AND METHODS

Partially stabilized yttria zirconium dioxide, zirconia (Starceram® Z-Al Med HD, H.C. Stark Ceramics, Laufenburg, Germany), three-dimensional rectangular specimens (10×10×2 mm^3) were fabricated in the Planmeca milling center (Planmeca, Helsinki, Finland). The specimens were ground from green-stage blocks, polished on both sides with 800 and 1200 grit silicon carbide paper (Struers, Copehagen, Denmark) and then sintered in a sintering furnace according to manufacturers' instructions. A total of 60 specimens were ultrasonically cleaned in distilled water and ethanol and air-dried and divided into 3 groups according to surface treatment and bonding procedures. These subgroups were then further divided into two groups according to hydrothermal aging (dry/thermocycling) (n=10/group). All the materials used in the study and the study groups with different surface treatments are presented in detail in Tables 1 and 2.

Air-borne particle abrasion

In the ABP group the "golden standard" of zirconia bonding procedures was used[9]. The specimens were air-borne particle abraded (Cojet, 3M Espe, Seefeld, Germany) with dialuminum trioxide particles (Korox, Beco, Bremen, Germany), having a particle size of 50 μm with a pressure of 250 kPa for 15 s. In the ABPR-S group, specimens were air-borne particle abraded with

Table 1 Detailed information about the study materials

Materials commercial name	Content*	Manufacturer	Lot No.
Starceram® Z-Al Med HD Color 800	ZrO_2>99.0 wt% Y_2O_3 5.15±0.2 wt% HfO_2<5.0 wt% Al_2O_3<0.3 wt% Fe_2O_3<0.2 wt% Na_2O<0.04 wt%	H.C.Starck, Laufenburg, Germany	50573391
Korox	Al_2O_3 50 μm	Beco, Bremen, Germany	1495741 0713
Rocatec soft	Al_2O_3 30 μm, modified with SiO_2	3M Espe, Seefeld, Germany	424975
Potassium hydrogen fluoride	KHF_2>99%	Honeywell International, Charlotte, NC, USA	H054A
Clearfil ceramic primer	10-MDP 3-MPS<5 wt% Ethanol>80 wt%	Kuraray, Tokyo, Japan	350009
Clearfil porcelain bond activator	3-MPS 40–60 wt% Hydrophobic aromatic dimethacrylate	Kuraray	9C0040
Panavia F 2.0	N-Methacryloyl-5-aminosalisylic acid Water Catalyst Accelerators	Kuraray	000060

* Information about material content is provided by the manufacturers.
10-MDP=10-methacryloyloxydecyl dihydrogen phosphate; 3-MPS=3-methacryloxypropyltrimethoxysilane

Table 2 Study groups of Starceram® zirconia with different surface treatments

Study group	Surface treatment	Conditioning	Adhesive cement
ABP	Air-borne particle abrasion Al_2O_3 50 µm, 250 kPa (Korox)	Zirconia primer (Clearfil Ceramic Primer)	Resin based luting cement (Panavia F 2.0)
ABPR-S	Air-borne particle abrasion with tribochemical silica, 30 µm 280 kPa (Rocatec Soft)	Silane (Clearfil porcelain bond activator)	Resin based luting cement (Panavia F 2.0)
ETC	Etching with KHF_2 (Potassium Hydrogen Fluoride)	Silane (Clearfil porcelain bond activator)	Resin based luting cement (Panavia F 2.0)

a tribochemical system of silica coated trialuminium trioxide particles with a particle size of 30 µm and an air pressure of 280 kPa for 15 s (Rocatec Soft, 3M Espe). The nozzle of the air-borne particle abrasion unit was kept at a 10 mm distance to the specimens. The specimens were ultrasonically cleaned after the air-borne particle abrasion.

Etching

In the ETC group the specimens were etched with potassium hydroxide difluoride (KHF_2) slurry, as described by Ruyter and co-authors[11]. In this method, fine ground KHF_2 crystal powder (Potassium Hydrogen Fluoride, Honeywell, USA) was mixed with distilled water (4.0 mg/mL) to a viscous slurry and spread on the surface of the zirconia specimens. The specimens were then placed on a metal plate and then heated in a preheated oven for 10 min at a temperature of 280ºC. After cooling the specimens at room temperature, the etched specimens were ultrasonically cleaned and air-dried.

Surface roughness

The surface roughness of the specimens was measured after surface treatment (air-borne particle abrasion or etching procedures) and ultrasonic cleaning (in distilled water and ethanol for 15 min) with non-contacting optical profilometer (Contour-GT-K1, Bruker, Billerica, MA, USA) and analyzed with a Bruker Vision 64 software (version 5.41, update 4, Bruker) with a lateral resolution of 0.36 µm. Three specimens per group were analyzed at two different magnifications, 5× and 20×. Artihmetric average roughness (R_a), root mean squared (R_q) and maximum valley depth (R_v) values (µm) were recorded.

Silanization and cementation

In the ABPR-S and ETC groups, the specimens were silanized with Clearfil Porcelain bond activator (Kuraray, Tokyo, Japan). In the ABP group MDP-monomer containing zirconia primer Clearfil Ceramic Primer (Kuraray) was applied to the specimen surface.

After the silanization procedure, adhesive cement stubs were applied to the center of the specimens with the help of a silicone mold. The diameter of the adhesive cement stub was 3.6 mm with a thickness of 4 mm, according to the adopted protocol of shear bond strength testing[19]. In all the groups, adhesive cement Panavia F 2.0 (Kuraray) was used and light cured according to the manufacturer's instructions for a minimum of 40 s. All the specimens were stored dry at room temperature 24 h before the bonding test and thermocycling.

Bonding test

The bonding test followed the adopted protocol of bond strength testing with the application of predominantly shear stress at the interface (SBS test)[19]. Half of the specimens (n=10) of each surface treatment group (ABP, ABPR-S and ETC) were tested dry and the other half of the specimens (n=10) were thermocycled in water for 6000 cycles at 5–55ºC. After thermocycling, the specimens were stored in distilled water (37ºC) for 36 h and then tested. The bond strength test was performed at room temperature by using a universal testing machine (model LRX, Lloyd instruments, Fareham, England). The specimens were placed in the holder of a testing machine and the bonded cement stub was loaded with a parallel knife-edge blade which was positioned to be in contact with the flat-ground surface of the holder and substrate. The interface of the ceramic and the cement was loaded until the fracture occurred. The crosshead speed of the blade was 1.0 mm/min. The shear bond strength was calculated by dividing the highest fracture force (N) with the area of the bonded cement (diameter 3.6 mm) and recorded in MPa using PC software (Nexygen, Lloyd Instruments).

Scanning electron microscopy

After the bonding test, all specimens were coated with gold (Bal-Tec SCD 050, Sputter coater) and the fracture surfaces were evaluated using a scanning electron microscope (SEM) (Princeton Gamma-tech X-ray microanalysis). Magnifications of ×120, ×500 and ×1,000 were used. The failure mode of the adhesive surface was defined (adhesive/cohesive). The surface topography of different treatments and plain zirconia after sintering without surface treatment was also evaluated by SEM with magnifications of ×1,000, ×2,500, ×5,000 and ×6,000.

Statistical analysis

The statistical analysis was conducted using SPSS Statistics (IBM, Armonk, NY, USA). The normality of the data was assessed using the using Shapiro-Wilk test. Descriptive statistics are presented as means, standard deviations, medians and interquartile ranges. The Kruskall-Wallis test was used to examine the differences in the surface roughness between different surface treatments. The Mann-Whitney *U* test was used to examine the effect of aging on the bond strength in each surface treatment group separately. Finally, the Kruskal-Wallis test was used to examine the differences in bond strength between surface treatment groups in both aging method groups (dry/thermocycled in water) separately. p-Values<0.05 were considered statistically significant.

RESULTS

The surface characteristics of plain zirconia surface after sintering and before roughening, and different study groups after sintering and surface treatments are seen in Figs. 1a–d. The images were taken with SEM at magnification of ×6,000. The mean and median

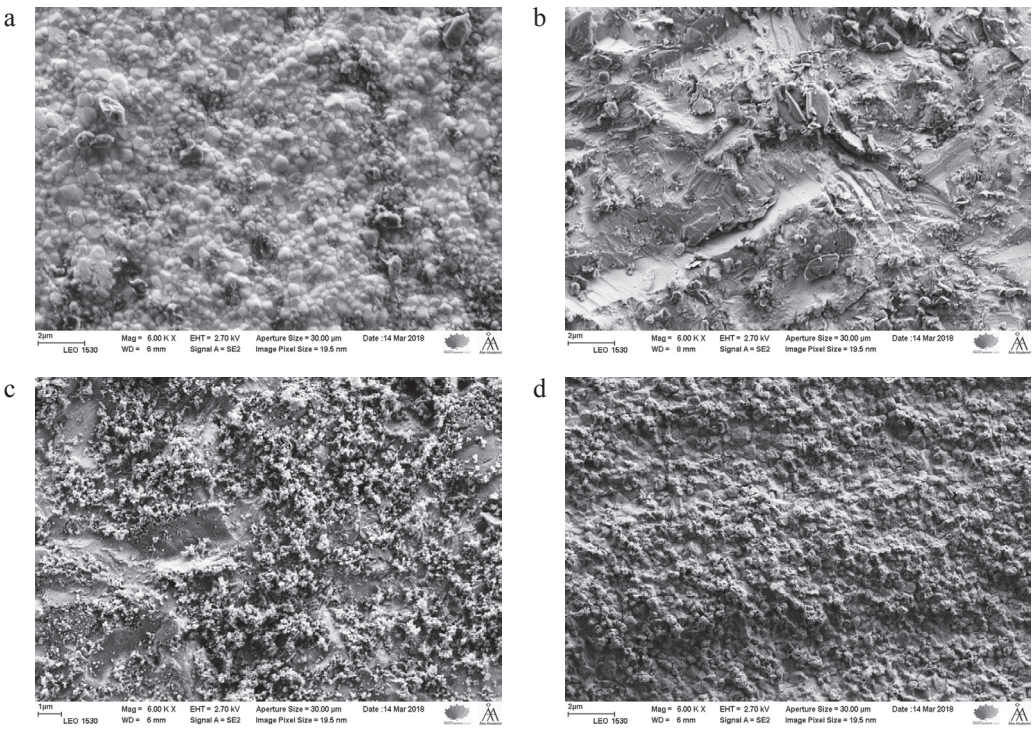

Fig. 1 The surface characteristics of plain zirconia surface after sintering and before roughening and different study groups after sintering and surface roughening with the scanning electron microscope at magnification of ×6,000: a) Zirconia material used in the study, before roughening, b) Air-borne particle abrasion (Group ABR), c) Tribochemical silica coating (Group ABPR-S), d) Etching with KHF_2 (Group ETC).

Table 3 The mean values (μm) (SD) and median values (μm) from surface roughness testing described with three different parameters: artihmetric average roughness (R_a), root mean squared (R_q) and maximum valley depth (R_v) values at two different magnifications. There was no statistically significant difference within these parameters between the different surface treatments tested with non-parametric test (Kruskall-Wallis).

	ABP						ABPR-S						ETC					
	5×			20×			5×			20×			5×			20×		
Unit	Ra (μm)	Rq (μm)	Rv (μm)	Ra (μm)	Rq (μm)	Rv (μm)	Ra (μm)	Rq (μm)	Rv (μm)	Ra (μm)	Rq (μm)	Rv (μm)	Ra (μm)	Rq (μm)	Rv (μm)	Ra (μm)	Rq (μm)	Rv (μm)
Mean (SD)	0.20 (0.01)	0.25 (0.01)	−1.30 (0.03)	0.29 (0.04)	0.35 (0.04)	−1.48 (0.14)	0.33 (0.08)	0.49 (0.2)	−8.09 (8.12)	0.42 (0.09)	0.52 (0.11)	−2.9 (1.84)	0.31 (0.06)	0.39 (0.08)	−4.87 (5.59)	0.34 (0.1)	0.43 (0.11)	−2.89 (1.41)
Median	0.20	0.25	−1.30	0.29	0.36	−1.49	0.33	0.43	−5.57	0.39	0.50	−1.87	0.34	0.42	−1.78	0.31	0.39	−2.16

surface roughness values measured with two different magnifications are seen in Table 3. Normality analysis showed that the surface roughness data was not normally distributed in all the subgroups. Non-parametric testing (Kruskall-Wallis) indicated no statistically significant differences in R_a, R_q or R_v parameters ($p>0.05$) between the groups.

The normality analysis of the results from the SBS test showed that the data was not normally distributed in all the subgroups. Therefore, nonparametric tests were used to compare the differences between the groups. The mean values and standard deviations of shear bond strength (MPa) as well as medians and interquartile ranges are presented in Table 4. In all the surface treatment groups shear bond strength decreased significantly when the specimens were thermocycled and stored in distilled water before the SBS test (Mann-Whitney-*U* test, $p<0.05$). For the specimens stored dry, there were no statistically significant differences between the different surface treatments in bond strength, varying on the level of 19.5–22.5 MPa (Kruskal-Wallis test, $p>0.05$). In thermocycled groups the SBS varied from 2.4–7.4 MPa. The Kruskal-Wallis test revealed statistically significant differences between ABP and ABPR-S groups ($p<0.05$) and between ABP and ETC groups ($p<0.05$) in bond strength, with the ABP treatment giving significantly lower SBS values.

Failure modes of the specimens were evaluated with SEM and are seen in Fig. 2. Most of the failures in groups ABP and ABPR-S were adhesive. In the group ETC most failures were adhesive/cohesive, especially after the thermocycle. Figures 3a–c illustrates the examples of different failure modes.

Table 4 The mean values (MPa) and standard deviations as well as medians and interquartile ranges (IQR) of bond strength testing (SBS) of the study groups according to different surface treatments and testing methods (dry/thermocycled in water). The statistical analysis was done with non-parametric tests using median SBS values.

Group		Mean SBS MPa (SD)	Median SBS MPa (IQR)*
Dry	ABP	19.5 (6.9)	20.9 (12.7, 26.4)[a1]
	ABPR-S	22.5 (4.2)	23.4 (20.1, 25.6)[a1]
	ETC	21.8 (4.2)	21.8 (18.6, 26.1)[a1]
Tc	ABP	2.4 (1.5)	2.1 (1.3, 3.7)[b3]
	ABPR-S	5.9 (4.1)	5.8 (2.9, 7.2)[b2]
	ETC	7.4 (5.4)	5.7 (3.5, 10.8)[b2]

*Different letters indicate statistically significant differences in the median SBS values based on the aging (dry *vs* thermocycle) ($p<0.05$, Mann-Whitney-*U* test) and different numbers indicate statistically significant differences in SBS values between the different surface treatment groups in both dry and thermocycling groups separately ($p<0.05$, Kruskall-Wallis test).

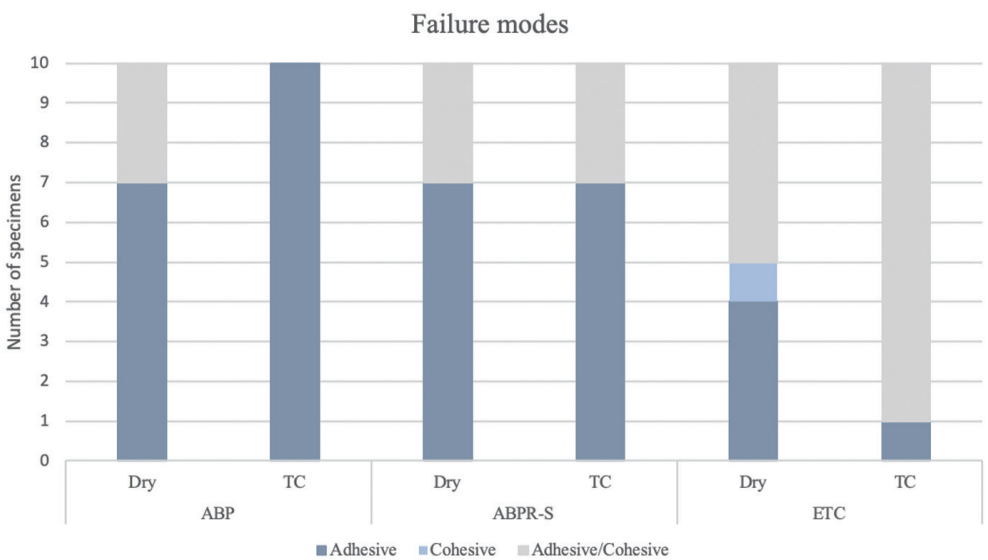

Fig. 2 The amount of different failure modes in different study groups after the shear bond strength test.

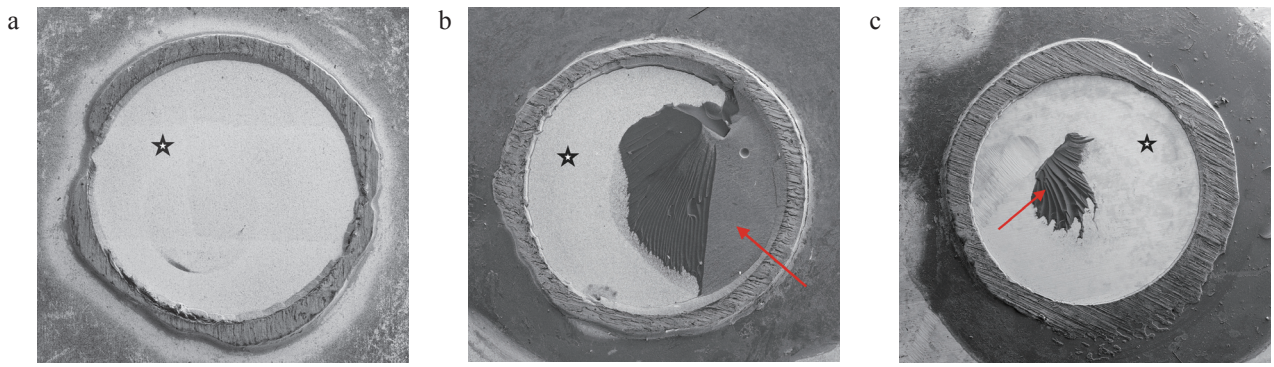

Fig. 3 Examples of failure modes of the study groups taken with SEM magnification of 50×: a) ABP with adhesive failure, b) ABPR-S with adhesive/cohesive failure, c) ETC with adhesive/cohesive failure. White area is exposed zirconia (star) and dark grey areas (arrow) are resin composite cement remnants.
The diameter of the exposed zirconia area is 3.6 mm.

DISCUSSION

This *in vitro* study was conducted in order to compare bond strength using a novel etching system with previously introduced and commonly clinically used surface roughening (air-borne particle abrasion or tribo-chemical coating) and chemical adhesion methods (silane and MDP-monomer containing primer) for resin based luting cements. The study hypothesis could be partially accepted since there were no statistically significant differences in shear bond strength between the different surface roughening methods tested when dry. However, thermocycling and water storage reduced the bond strength significantly in all study groups and after thermocycling, the ABP group had statistically significantly lower SBS compared to ABPR-S and ETC groups.

The visual inspection with a scanning electron microscope of the roughened surfaces showed clear differences on the surface structure of the groups. Compared to the zirconia surface without any treatment, the air-borne particle abraded surface had a coarse rough surface. The tribochemically treated surface as well as etched surfaces showed more fine-grained surface structure. All the surface treatments increased the visual surface micro roughness and allowed micromechanical interlocking of the cement. KHF_2 etching was done in order to imitate the surface of hydrofluoric acid etched glass ceramic and to gain more micromechanical retention. As suggested in the literature, the profile of the roughness, *i.e.* maximum valley depth (R_v) vs. maximum peak height (R_p) of hydrofluoric acid etched glass ceramics is greater when the etching time is longer[20]. The surface has deeper vertical deviation which enhances micromechanical bonding that is even to tensile stress perpendicular to the substrate surface. Lower R_v of air-borne particle abraded zirconia could improve resistance mainly against shear types of stress.

A non-contacting optical profilometer (Contour-GT-K1, Bruker) with a lateral resolution of 0.36 µm was used to measure surface roughness values, which did not differ between the groups. However, there could have been some differences in surface characteristics at the nano-level as well. A nano-level roughness measurement based on surface area would have given more accurate information on the surface characteristics[21] and should perhaps be considered in future studies.

In a recent study the novel etching technique developed by Ruyter and colleagues was investigated, and it was shown that etching with KHF_2 or $NH4HF_2$ leads to higher shear bond strengths than air borne particle abrasion with alumina particles[18]. The SBS with etched groups varied between 4.1–5.6 MPa compared to the air-borne particle abraded group that had a shear bond strength of 1.5 MPa. With the KHF_2 or $NH4HF_2$ etching technique, the surface roughness was lower than with air-borne particle abrasion and it was discussed that the bonding ability could have been enhanced by the better wettability of the surface. However, in that study they did not use the MDP-monomer containing primer, which was shown to be substantial for the successful clinical outcome of zirconia bonding[22]. In the present study the SBS of all the groups varied between 19.5–22.5 MPa when tested dry and 2.4–7.4 MPa when tested after thermal cycling. No differences were seen regarding the roughening method among the dry tested groups. After thermocycling the ABP group showed lower SBS compared to ABPR-S and ETC groups. In the study of Ruyter and co-workers the bond strength values with etched specimens varied between 31.0–42.7 MPa[11]. Although, these values are not comparable to studies with shear bond strength testing, because in their study a different testing method was used.

The role of MDP-monomer in the bond strength of zirconia-to-resin cement has been addressed in several studies[9,22-24]. In this study air-borne particle abrasion and MDP-monomer containing primer was used as a control group. For the ABPR-S and ETC groups a silane coupling agent was used to increase the wettability of the surface in order to achieve the best possible

micromechanical bond strength. A primer including both MDP and silane coupling agent or separate bonding agend was not used, because the aim was to investigate the effect of the surface roughening method itself. Visual inspection of ABPR-S and ETC groups showed fine-grained surface structure compared to the MDP group, which also supports the method to increase surface wettability. Chemical reaction between the ETC surface and silane coupling agend was not expected. In the ABPR-S group SBS was based on micromechanical retention and chemical reaction between silane coupling agents and silica coated alumina particles on the surface of the material. In dry tested groups, comparable bond strength values could be achieved also without MDP-monomer. In previous studies there have been concerns about the hydrolytic stability of the interfacial adhesion and bond strength between zirconia and resin composites, when silane coupling agents[14] or MDP-monomers[17] are used. Finally, it should be noted that in the present study there were 10 specimens per group and in some of the groups the standard deviations of SBS values were relatively high. With a larger amount of specimens per group the the outcome and the methodology could have been better validated.

The failure modes after the SBS test were adhesive in the air-borne particle abraded groups ABP and ABPR-S, whereas in the group of KHF_2 etched specimens, the failures were mainly adhesive-cohesive. It has been suggested before that higher microshear bond strength indicates more cohesive cement failures, which could be due to increased contact area and improved chemical interaction between the zirconia ceramic and resin cement after certain surface treatment methods[25]. Mainly cohesive failures were seen also in KHF_2 etched zirconia after the tensile bond strength test[26]. In the present study, there was a variation in failure modes although not all the groups presented statistically significant differences in bond strength.

In the present study the 36 h water storage after thermocycling was used to slightly prolong the aging of the bonding, although it is known that complete water saturation of the specimens of this size takes several weeks[27]. The primary aim of this study was to inspect the effect of different surface treatments to SBS and therefore only one aging method was chosen. There was a large drop in the SBS after thermocycling and water storage, although in the thermocycled groups, the SBS values were comparable to previous studies with similar testing conditions[8,18]. It has been shown that the aging conditions and aging time affect the bond strength of zirconia ceramics especially if the surfaces are not roughened with air-borne particle abrasion. This is because the bonding is predominantly based on chemical adhesion rather than micromechanical retention[16,28]. However, there are also studies where hydrothermal aging has not been shown to affect to the bond strength[29]. The reliability of zirconia bonding is still low and the bonding procedures are technique sensitive when they are performed under clinical conditions. Continuous material and bonding system development is needed to obtain more reliable bonding systems.

Within the limitations of this study, following can be concluded: The novel etching technique with KHF_2 did not provide better bonding capacity in the SBS test to the previously introduced surface roughening and chemical adhesion methods. Thermocycling and water storage decreased the bonding strength, a result which was statistically significant, with all of the tested methods.

ACKNOWLEDGMENTS

The authors are grateful to biostatistician Paula Bergman, Univesity of Helsinki, for statistical consultation.

CONFLICT OF INTEREST

The authors declare no conflicts of interest.

REFERENCES

1) Larsson C, Vult von Steyern P. Ten-year follow-up of implant-supported all-ceramic fixed dental prostheses: a randomized, prospective clinical trial. Int J Prosthodont 2016; 29: 31-34.
2) Tanner J, Niemi H, Ojala E, Tolvanen M, Närhi T, Hjerppe J. Zirconia single crowns and multiple-unit FDPs –an up to 8-year retrospective clinical study. J Dent 2018; 79: 96-101.
3) Pjetursson BE, Valente NA, Strasding M, Zwahlen M, Liu S, Sailer I. A systematic review of the survival and compication rates of zirconia-ceramic and metal-ceramic single crowns. Clin Oral Implants Res 2018; 29 s16: 199-214.
4) Sailer I, Makarov NA, Thoma DS, Zwahlen M, Pjetursson BE. All-ceramic or metal-ceramic tooth-supported fixed dental prostheses (FDPs)? A systematic review of the survival and complication rates. Part I: single crowns (SCs). Dent Mater 2015; 31: 603-623.
5) Kern M, Passia N, Sasse M, Yazigi C. Ten-year outcome of zirconia ceramic cantilever resin-bonded fixed dental prostheses and the influence of the reasons for missing incisors. J Dent 2017; 65: 51-55.
6) Rathmann F, Bömicke W, Rammelsberg P, Ohlmann B. Veneered zirconia inlay-retained fixed dental prostheses: 10-year results from a prospective clinical study. J Dent 2017; 64: 68-72.
7) Thoma DS, Sailer I, Ioannidis A, Zwahlen M, Makarov N, Pjetursson BE. A systematic review of the survival and complication rates of resin-bonded fixed dental prostheses after a mean observation period of at least 5 years. Clin Oral Implants Res 2017; 28: 1421-1432.
8) Matinlinna JP, Heikkinen T, Özcan M, Lassila LVJ, Vallittu PK. Evaluation of resin adhesion to zirconia ceramic using some organosilanes. Dent Mater 2006; 22: 824-831.
9) Kern M. Bonding to oxide ceramics —laboratory testing versus clinical outcome. Dent Mater 2015; 31: 8-14.
10) Salem R, Naggar GE, Aboushelib M, Selim D. Microtensile bond strength of resin-bonded hightranslucency zirconia using different surface treatments. J Adhes Dent 2016; 18: 191-196.
11) Ruyter EI, Vajeeston N, Knarvang T, Kvam K. A novel etching technique for surface treatment of zirconia ceramics to improve adhesion of resin based luting cements. Acta Biomater Odontol Scand 2017; 3: 36-46.
12) Murakami T, Takemoto S, Nishiyama N, Aida M. Zirconia surface modifications by a novel zirconia bonding systems and its adhesion mechanism. Dent Mater 2017; 33: 1371-1380.

13) Zhuravlev LT. Concentration of hydroxyl groups on the surface of amorphous silicas. Langmuir 1987; 3: 316-318.
14) Heikkinen TT, Matinlinna JP, Vallittu PK, Lassila LV. Long term water storage deteriorates bonding of composite resin to alumina and zirconia. Short communiucation. Open Dent J 2013; 7: 123-125.
15) Pitta J, Branco TC, Portugal J. Effect of saliva contamination and artificial aging on different primer/cement systems bonded to zirconia. J Prosthet Dent 2018; 119: 833-839.
16) Le M, Larsson C, Papia E. Bond strength between MDP-based cement and translucent zirconia. Dent Mater J 2019; 38: 480-489.
17) Chen C, Chen Y, Lu Z, Qian M, Xie H, Tay FR. The effects of water on degradation of the zirconia-resin bond. J Dent 2017; 64: 23-29.
18) Akazawa N, Koizumi H, Nogawa H, Kodaira A, Burrow MF, Matsumura H. Effect of etching with potassium hydrogen difluoride and ammonium hydrogen difluoride on bonding of a tri-n-butylborane initiated resin to zirconia. Dent Mater J 2019; 38: 540-546.
19) ISO Standard No. 29022. Adhesion –Notched-edge shear bond strength test, Geneva: International Organization for Standardization; 2013 p. 1-11.
20) Ramakrishnaiah R, Alkheraif AA, Divakar DD, Matinlinna JP, Vallittu PK. The effect of hydrofluoric acid etching duration on the surface micromorphology, roughness, and wettability of dental ceramics. Int J Mol Sci 2016; 17(6). pii: E822.
21) Wennerberg A, Albrektsson T. Suggested guidelines for the topographic evaluation of implant surfaces. Int J Oral Maxillofac Implants 2000; 15: 331-344.
22) Lümkemann N, Eichberger M, Stawarczyk B. Different surface modifications combined with universal adhesives: the impact on the bonding properties of zirconia to composite resin cement. Clin Oral Investig 2019; 23: 3941-3950.
23) Kitayama S, Nikaido T, Takahashi R, Zhu L, Ikeda M, Foxton RM, *et al.* Effect of primer treatment on bonding of resin cements to zirconia ceramic. Dent Mater 2010; 26: 426-432.
24) Nagaoka N, Yoshihara K, Tamada Y, Yoshida Y, Van Meerbeek B. Ultrastructure and bonding properties of tribochemical silica-coated zirconia. Dent Mater J 2019; 38: 107-113.
25) Ali N, Safwat A, Aboushelib M. The effect of fusion sputtering surface treatment on microshear bond strength of zirconia and MDP-containing resin cement. Dent Mater 2019; 35: e107-112.
26) Sagen MA, Kvam K, Ruyter EI, Rønold HJ. Debonding mechanism of zirconia and lithium disilicate resin cemented to dentin. Acta Biomater Odontol Scand 2019; 5: 22-29.
27) Pastila P, Lassila LVJ, Jokinen M, Vuorinen J, Vallittu PK, Mäntylä T. Effect of short-term water storage on the elastic properties of some dental restorative materials –A resonant ultrasound spectroscopy study. Dent Mater 2007; 23: 878-884.
28) Aboushelib MN, Ragab H, Arnaot M. Ultrastructural analysis and long-term evaluation of composite-zirconia bond strength. J Adhes Dent 2018; 20: 33-39.
29) Ruales-Carrera E, Cesar PF, Henriques B, Fredel MC, Özcan M, Volpato CAM. Adhesion behavior of conventional and high-translucent zirconia: effect of surface conditioning methods and aging using an experimental methodology. J Esthet Restor Dent 2019; 31: 388-397.