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SYSTEMATIC REVIEW

Is there a potential for durable adhesion to zirconia restorations? A systematic review

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ABSTRACT

Statement of problem. With a number of zirconia ceramic materials currently available for clinical use, an overview of the scientific literature on the adhesion methods and their potential influence is indicated.

Purpose. The purpose of this systematic review was to classify and analyze the existing methods and materials proposed to improve adhesion to zirconia surfaces.

Material and methods. The current literature of in vitro studies examining the bond strength on zirconia ceramics, including clinical studies from 1998 until 2014, was analyzed. A search of the English language literature was undertaken using MEDLINE and PubMed, and a hand search was made for any relevant research paper from the library of a dental school. Papers evaluating only alumina restoration bond or ceramic-zirconia bond were excluded.

Results. A total of 134 publications were identified for analysis. Different adhesive techniques with different testing methods were reviewed. Results were difficult to compare in that the parameters varied in each research protocol.

Conclusions. Airborne-particle abrasion and tribochemical silica coating are reference pretreatment methods. Adhesive monomers are necessary for chemical bonding. Surface contamination and aging have negative effects on adhesion to zirconia. Many factors influence each combination of zirconia material, such as surface treatment, adhesive medium, and aging conditions. Laboratory studies should be confirmed by clinical trials. (J Prosthet Dent 2016;115:9-19)

To achieve the best possible bond quality for the long-term survival of a prosthesis, the intaglio should adhere to the luting agent.[1,2] Zirconia cores are almost unaffected by any processing because of their high hardness and crystallinity.[1] Because zirconia is not etchable, the advantage of stronger adhesion using resin cements may be lost.[2,3] However, under appropriate conditions, resin cements provide stronger bonding for zirconia restorations with better mechanical properties than conventional cements.[4-12]

Particularly in restorations in which mechanical retention by the abutment is limited, reliable bonding of resin cements with zirconia improves the application limits, reduces microleakage, and increases retention.[13,14] In single retainer ceramic resin-bonded partial fixed dental prostheses (FDPs) and in inlay-retained FDPs, improved adhesion would minimize the possibility of decementation.[15,16] In single zirconia crowns, the loss of retention has been shown to be significantly higher than that for other etchable ceramic crowns.[17,18]

Most information about adhesion has come from laboratory studies, and their conclusions may well be useful in guiding randomized clinical trials (RCTs).[19] As-produced zirconia surfaces show low bond strengths even with adhesive resin cements.[20] Laboratory experiments have limitations, and the results of different techniques are not always comparable.[21] Each different zirconia material has different surface features and internal structure, grain size, shape, composition, and hardness so that the effect of any surface treatment and the consequent bond strength with different materials may vary,[22] making it inappropriate to generalize findings from one type of material to another.[3] However,

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Clinical Implications

No universally accepted protocol exists for long-lasting and biologically safe zirconia cementing.

preliminary clinical observations show most common and simple bonding methods to be reliable.[23] A list of all available treatment methods for surface preparation is shown in Figure 1. The purpose of this systematic review was to classify and analyze the existing methods proposed to improve adhesion to zirconia surfaces.

MATERIAL AND METHODS

A preliminary search using MEDLINE and PubMed with the keywords "zirconia and bond," "zirconia and abrasion," "zirconia and lasers," "zirconia and primers," "zirconia and silanes" helped to classify the most popular surface treatments. The material reviewed consisted of mainly laboratory studies and a small number of systematic reviews and RCTs for zirconia restoration bonding. Publications containing characterizations of zirconia materials after surface treatments were also included. Further information on each technique and material was found by hand searching a university library for any relevant papers. All articles reporting on only alumina materials were excluded. A total number of 134 publications from 1998 to September 2014 were reviewed.

Airborne-particle abrasion

Airborne-particle abrasion (APA) can be applied to metals and ceramics[24] and hard dental tissues (enamel, dentin)[25] and has also been proposed for roughening the surface of zirconia as a way of increasing mechanical interlock and total contact area.[26-30] The variable parameters in APA with alumina are grain size (25 to 250 μm), propulsion pressure (0.05 to 0.45 MPa), distance (5 to 20 mm) from the nozzle to the specimen, and time of APA (5 to 30 seconds).[31,32]

The micromechanical retention of zirconia surfaces treated by abrasion with small (25 μm, 50 μm) or larger grains (110 μm) was not significantly different,[14,33,34] despite the different surface roughness produced.[35] Although a larger grain size creates a rougher zirconia surface,[35] bond strength is not significantly influenced.[14,36] Also, APA increases surface roughness without improving micromechanical retention.[30] Other researchers have observed smoother surface topography but improved bond strength with resin cements after APA with alumina grains (50 μm).[27,37,38]

The effect of APA on surface roughness depends also on the type of zirconia material.[22] Removing the waste alumina from the surface seems particularly important.[39] The kinetic energy of a grain as it collides with the surface is directly proportional to the mass of the granule, which in turn increases with the cube of the diameter.[40] Reducing the pressure during APA does not seem to affect long-term bond strength when adhesive surface activators are used (adhesive primers).[41] APA increases surface energy and reduces organic contaminants, thus improving the wettability of the surface.[42] The relative benefits and the extent of the influence on the bond strength of APA or tribochemical silica coating (TBC) are listed in Table 1.

The use of APA raises 2 main concerns: the possible creation of surface microcracks and the activation of phase transformation from tetragonal to monoclinic (t→m) at the surface and subsurface, which in turn can reduce the mechanical properties of the material.[43-45] To balance the effect of microcracks generated by aggressive APA, surface compressive strength is needed.[46] Nevertheless, counteracting the strength reduction of the microcracks is not sufficient.[44] For this reason, manufacturers suggest heating after APA to reverse the (m→t) conversion[47] or using APA before the final sintering.[48,49] Some manufacturers do not recommend its use with alumina grain up to 50 μm.[50,51] Significant phase conversion (t→m) appears to be caused by aggressive APA increasing the monoclinic phase.[52]

Alumina grit coated with silica was used to increase silicon in the surface of the zirconia and improve the bond with the resin bisphenol A glycidyl methacrylate or 10-methacryloyloxydecyl dihydrogen phosphate (MDP)-based cements.[36,53,54] Most researchers agree that this technique is better than simple APA,[26,36,55,57]especially when followed by silanization, or, at least, produces similar bond strengths.[56] Finally, no clear benefits were observed in the use of APA to enhance zirconia core-ceramic bond strength.[58,59] Table 2 uses selected articles to show the size of the effect of APA and TBC on bond strength.

Grinding with disks and diamond rotary instruments

The main disadvantage of grinding methods is again the possible creation of microcracks in the surface.[43] The high hardness of zirconia necessitates grinding with coarse diamond rotary instruments (120 to 200 μm grain size).[60] Previously, a coarse-grained diamond grinding method had been tested, producing a rougher surface than other techniques and improved bond strength but was not acceptable because it is an aggressive method that can induce microcracks and cause damage to zirconia surfaces.[61,62] Grinding conditions also seem important in that wet grinding with a 91-μm diamond wheel did not dramatically diminish flexural strength.[63] Grinding tests with 100-μm diamond rotary instruments on 3 different zirconia materials showed that in only 1 case was roughness significantly increased.[45]

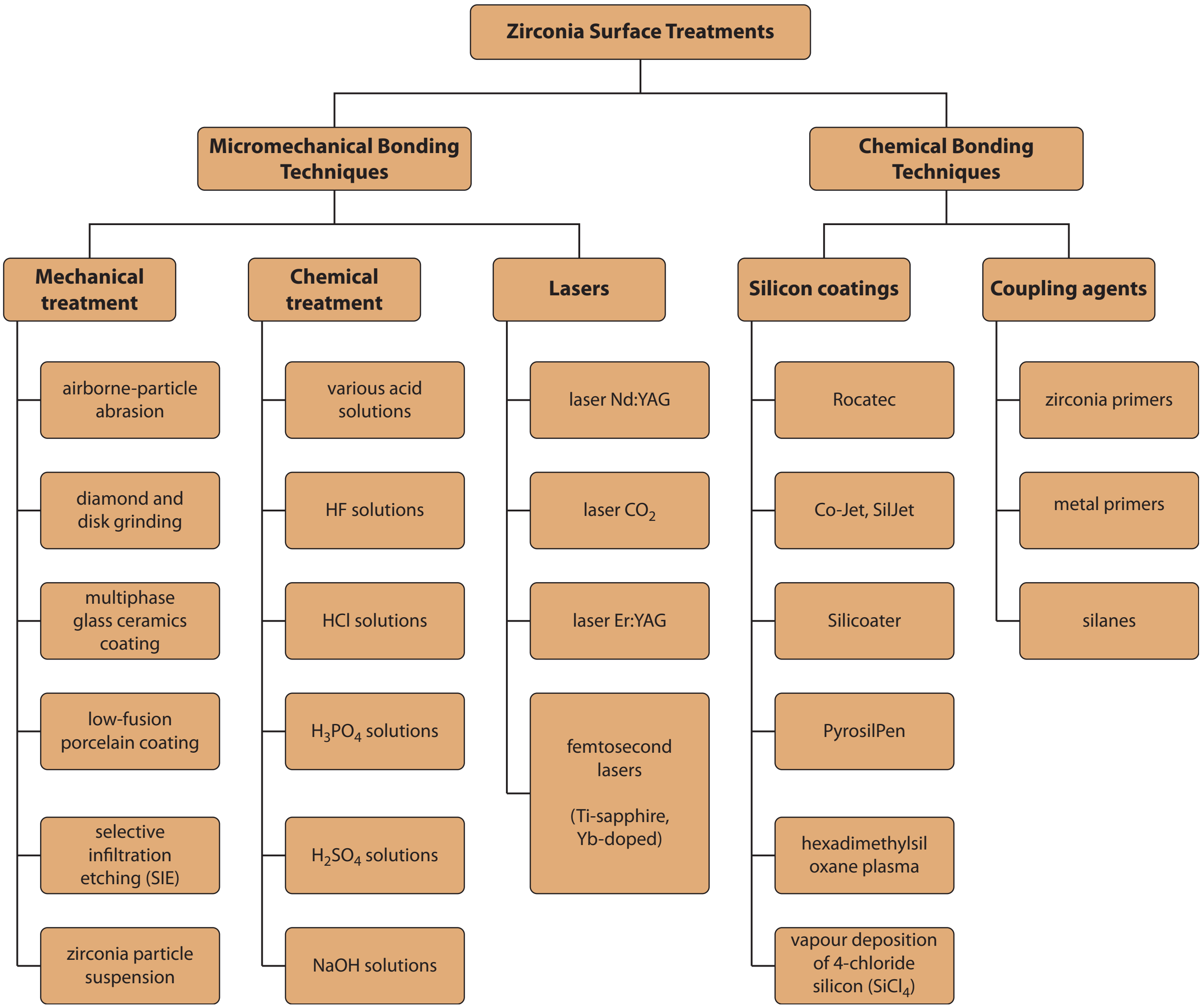

Figure 1. Summary of zirconia surface treatments before cementation.

Other techniques for increased roughness

In an effort to increase surface porosity, coatings of low-melting temperature porcelain micropearls and selective infiltration etching (SIE) have been tested.[64-66] Surface silicon allowed silanization before bonding and multiphase ceramic layers (lithium disilicate glazing) also yielded encouraging results.[47,67]

In the case of SIE, a smooth surface is transformed into a highly retentive one,[67,68] which demonstrates better bond strength than APA methods,[67,69] even after 2 years of artificial aging.[70,71] Nobelbond (Nobel Biocare) is a similar technique in which the fusion product is a porous ceramic coating composed of zirconia powder slurry.[50,72,73] The superiority of these special techniques over other surface treatments is shown in Table 1 using extracts from selected articles.

Effect of chemical agents

The extremely high crystallinity of the zirconia core, with a glassy phase <1%, and a low content of silicon dioxide, makes it practically impervious to treatment with hydrofluoric acid (HF), with no improvement in bond strength.[30,31,54,61] The slight increase in the bond strength of zirconia with resin cements after applying HF was not statistically significant.[74,75] HF reacts with silicon dioxide (SiO_2), and the silicic derivatives are water soluble and can be flushed away,[37] leaving micropitting on the surface of the ceramic.[76] Attempts at etching with a higher concentration of HF and longer application time (40% HF for 210 seconds) showed improved shear bond strengths compared with the control and metal primer groups; this needs further investigation.[77]

Table 1. Studies comparing surface treatment techniques influencing micromechanical retention

Author	Material Resin/Cement/Primer	Surface Treatment	Reference Treatment	Modified vs Reference Bond Strength/MPa(SD)	Effect of Method on Bond Strength
Blatz 2010	Katana, Noritake/G-Cem (4-META)	APA 50 μm 2.8 bar, 12 s 10 mm	Untreated	22.4 vs 7.9	+283% SBS
Kern 1998	BCE Special Ceramics/Estiseal LC/Twinlook	APA 110 μm 2.5 bar, 13 s and TBC 110 μm	APA 110 μm,2.5 bar,13 s	29.0 (4.6) vs 14 (2.6)	+207% TBS
Tsukakoshi 2008	Nikkato/Rely X ARC 3M ESPE	Rocatec system 2.8 bar 10 s 10 mm/ESPE-Sil	Polishing #600 SiC	50.1 vs 5.4	+988% SBS
Abouselib 2007	Cercon Base, DeguDent	SIE/Panavia 2.0/1 month storage	APA /Panavia 2.0	52.2 vs 32.5	+147% MTBS
Derand 2005	Procera Zircon, Nobel Biocare	Micropearls of low fusing porcelain/silane/Variolink II	RF plasma treatment/silane/ Variolink II	18.4 (3.6) vs 5.3 (0.7)	+347% SBS
Kitayama 2009	Cercon Base, DeguDent	INT (internal coating technique)/Superbond C&B/Silane	APA/ Superbond C&B/Silane	18.9 (1.4) vs 12.7 (1.5)	+148% TBS
Paranhos 2011	Lava, 3M ESPE	Nd:YAG laser/Clearfil ceramic primer/Panavia 2.0	No treatment/ Clearfil ceramic primer/Panavia 2.0	14.9 (1.88) vs 4.65 (1.31)	+303% SBS
Foxton 2011	Procera Zircon, Nobel Biocare	Er:YAG laser/ Variolink II/ 6 m storage	APA 53 μm 2.5 bar 15 s 10 mm/Variolink II/6 m storage	8.3 (1.15) vs 8.97 (2.76)	-10.8% SBS
Akyil 2010	Copran Zircon Blank, Whitepeaks Dental Systems GmbH	Laser CO_2/Clearfil ceramic primer/Clearfil Esthetic cement	APA 110 μm 2.8 bar 15 s 10 mm/ Clearfil ceramic primer/Clearfil Esthetic cement	22.35 (6.13) vs 23.46 (2.77)	-4,8% SBS

SBS, shear bond strength; TBS, tensile bond strength; APA, airborne-particle abrasion.

Table 2. Selected shear bond strength tests with thermocycling

Author/ Researcher Year	Zirconia Material	Surface Treatment/ Resin Cement/ Primer	Best Method in MPa (SD)	Thermocycling (TC)/ Water Storage	No. of Specimens/Control Group
Blatz 2004	Procera AllZirkon	APA 50 μ/Rely X	15.45 (3.79)-25.15 (3.48)	3-180 d and TC 12<ts>000 c	20/group/NO
Jenvicar 2010	TZ-3YB-E Tosoh		27.44 (3.23)	1 d-TC 12<ts>000c	10/group/YES
Liu 2013	Cercon, DeguDent	Zirconia coating/	12.5 (2.0)-16.0 (2.4)	1-30 d 3000-6000 TC	20/group/YES
Matinlinna 2013	Procera AllZircon, Nobel Biocare	APA and SC/silane ACPS/Rely X ARC	16 (2.5)-11.7 (2.3)	Dry- TC 6000c	12/group/NO
Moon 2011	Rainbow, Dentium	APA 70 μ before sintering/SuperBond C&B	19.69 (3.7)	1 d & TC 5000c	10/group/YES
Ozcan 2008	Lava Y-TZP, 3M ESPE	APA 50 μ/Panavia F2.0	9.6 (4.1)-0	Dry- 6000TC	10/group/NO
Phark 2009	Procera Zirconia, Nobel Biocare	Nobel Bond /Panavia 2.0	20.01 (3.45)-12.2 (2.45)	3- 90 d & 20000c TC	10/group/YES
Qeblawi 2010	ZirCAD, Ivoclar Vivadent AG	CoJet 30 μ/Monobond-S/Multilink Automix	30.9 (4.6)	10min-90 d & TC 6000c	12/group/YES
Tanaka 2008	Katana, Noritake	Rocatec Junior 30 μ/ESPE Sil/Epricode/Rely X ARC	48.24 (5.02)-50.81 (8.22)	1 d-15 d & TC 10<ts>000c	10/group/NO
Yoshida 2006	Shinagawa Fine Ceramics Co, Ltd	MDP primer/Zirconate coupler/Clapearl DC	46 (1.1)-57.6 (5.4)	1 d- 10<ts>000c TC	10/group/YES
Yun 2010	Rainbow Dentium	APA 90 μm/Alloy primer/Panavia F 2.0	16.7 (2.0)	1 d & 5000c TC	10/group/NO

ACPS, acryloyloxypropyltrimethoxysilane; APA, airborne-particle abrasion.

A similar procedure used for etching the metal wings of resin-retained fixed restorations was tested on zirconia and created a rougher surface. An experimental hot hydrochloric acid (HCl) solution significantly increased roughness, basically a controlled corrosion process.[65,78] The application of an HCl and Fe_2Cl_3 solution for 30 minutes enhanced the bond better than APA.[69] Sulfuric acid in solution with hydrogen peroxide (H_2O_2) (Piranha solution) appeared to have a positive effect on the bonding of zirconia with resin cements.[79] Hot acid etching with combinations of highly corrosive acids (HNO_3, H_2SO_4, and HF) improved both initial bond strengths and durability.[80]

Effect of lasers

Several types of lasers for cutting hard dental substances have been used by researchers to improve zirconia bonding capacity.[22,81,82] A neodymium-doped yttrium aluminum garnet (Nd:YAG) laser improved roughness and bond strength,[83-87] but the point of application left a silver spot[85] or greatly increased the monoclinic phase at the surface (26.5% and 30.5%).[87] A carbon dioxide (CO_2)

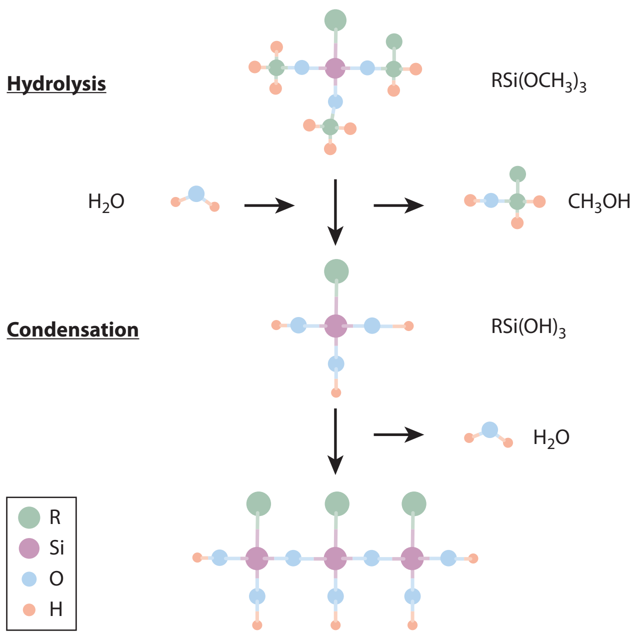

Figure 2. Hydrolysis of alkoxysilanes. (Adapted from Kato H, Matsumura H, Tanaka T, Atsuta M. Bond strength and durability of porcelain bonding systems. J Prosthet Dent 1996;75:163-8.)

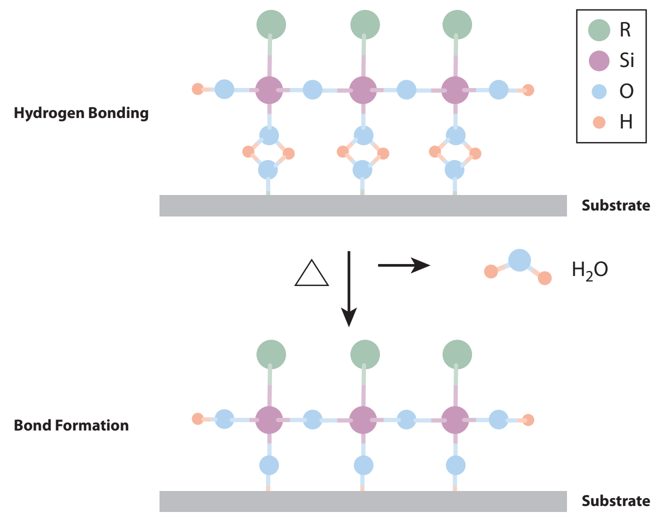

Figure 3. Bonding to inorganic surface. (Adapted from Kato H, Matsumura H, Tanaka T, Atsuta M. Bond strength and durability of porcelain bonding systems. J Prosthet Dent 1996;75:163-8.)

laser is suitable for ceramics because its emission wavelength (2.3 to 10.6 μm) is absorbed by ceramics. Improved bonding was found after this laser application at a setting of 3 W and 4 W with various settings.[75,82] At 4.5 W for 60 seconds, increased roughness and deep grooves were observed.[88] Ural et al,[89] while measuring the effects at power settings from 2 to 5 W (2, 3, 4, 5 W) observed that shear bond strength was improved at 2 W and negatively affected at 5 W. In a recent study, a CO_2 laser improved both roughness and the zirconia-porcelain bond.[90]

An erbium-doped yttrium aluminum garnet laser (Er:YAG) laser had been used for various clinical uses in operative dentistry,[91] and its action on high-strength ceramics had been studied extensively.[22] At high settings (600 mJ), extensive destruction of the material occurred, but increased roughness was observed; at low settings (200 or 400 mJ), the results were similar to those of airborne-particle abrasion.[22] At different settings (150 mJ, 1 W, low power for 20 seconds), Er:YAG seemed to improve bond strength.[86,92] According to Akyil et al,[83] power set at 2 W produced similar roughness to airborne-particle abrasion, with a better bond strength than the control group. Irradiation time appeared to play an important role.[83,93] Demir et al[94] also considered that applying Er:YAG at 400 mJ can be an alternative to APA with 110 μm alumina. This laser causes structural changes limited to the outer surface of the material.[95,96] Researchers have tested high-speed pulse lasers (femtosecond lasers) for surface treatment with promising results.[97-99] The influence of laser treatments is shown in Table 2.

Factors influencing chemical bonding

In general, silanes increase the wetting capacity of an inorganic surface, allowing a better flow of a resin cement across the surface and appear to enhance the micromechanical retention with low-viscosity resin cements. [43,100-104] The exact mechanism by which silanes link to 2 different substrates is complicated[100] (Figs 2, 3).

Silanes react with the zirconia powder in humid air or water to form Si-O-Zr linkages and stabilize t-phase.[101,105] They can be used alone or in combination with other surface treatments to increase bonding ability with resin cements.[64,68] In combination with the Rocatec Plus (3M ESPE AG) technique, the silane γ-MPS gave similar results to 3-acryloyloxypropyltrimethoxysilane (ACPS).[106] Silanes have been tested in combination with phosphates and phosphate methacrylates, and appear to perform better than conventional γ-MPS.[107] Nevertheless, a combination of γ-MPTS silane and 10-MDP primer reduced bond strength.[108] The action of silanes in combination with MDP provides reliable bonds.[109,110]

Plasma oxyfluoride has been used to coat the zirconia surface with a layer (1 to 3 nm) of zirconium oxyfluoride (ZrO_xF_y)[111,112] and significantly increased bond strength when combined with silane and resin cements containing MDP. Other coupling agents such as itaconic acid, oleic acid, and 2-OH-ethyl-methyl methacrylate were tested in a comparison with 2 silanes (ACPS and y-MPS) and appeared to be as effective as the silanes.[113]

Other surface coatings

TBC is similar to airborne-particle abrasion, except that the aluminum oxide is coated with silica.[57,114] Silicon concentration at the surface increases significantly,[103,114,115] but surface cleaning in an ultrasonic bath destroys this effect.[115,116] Increasing the pressure increased the roughness, the number of particles in contact with the surface, and the amount of silicon and eventually improved the bond strength.[117] In each case, the use of TBC increased the bond strength with zirconia, which has led to it becoming the reference method in contemporary research.[114,117,118] Another technique for creating chemical bond is silicoating (Silicoater; Heraeus Kulzer GmbH), which is based on the pyrolytic deposition of silicon to form an SiO_x-C coating with a thickness of 0.1 μm.[76] This surface can then be silanated to provide stronger bonds with metal and resin cements.[119,120] A similar technique is PyrosilPen Technology (PyrosilPen; SurA Instruments). Nevertheless, the results with zirconia ceramics have fallen short of expectations because of the extremely smooth surface and the inability to create a bond between silicon and zirconium.[14]

Surface treatment by plasma spraying hexamethyldisiloxane produced a thin (<1 μm) siloxane coating.[64] In molecular vapor deposition, zirconia specimens are exposed to 1-chloro silicide gas ($SiCl_4$) in the presence of water vapor for 15 minutes and produce an activated siliconized surface.[111,121] Nano-alumina coating on the surface of zirconia appears to improve the bond and is a simple and nondestructive method.[72] The coating of surfaces with zirconia ceramic glazes (glaze-on technique) gave improved values in shear tests, with the main disadvantage being a large thickness (120 μm) coating.[122] Zirconia particle deposition using a milling residue suspension seems promising and effective as airborne-particle abrasion.[44,123] A solid-gel process (sol-gel) is impractical because it takes many hours to create a silicate network in the surface (24 to 141 hours).[124] Nevertheless, when compared with conventional TBC, this technique gives the same shear bond strength, higher silicon content, and better durability.[125] Recently, alternative coatings of zirconia surfaces with fluorapatite-leucite glaze or salt glaze have proved unsatisfactory. Aggressive acid etching has produced similar bonds to conventional methods.[126] Another tested treatment is the addition of color modifiers to the mass of the zirconia material; although this process alters the zirconia/oxygen ratio of the surface and other surface characteristics, the bond strength with resin cements was not affected.[38]

Zirconia and metal primers

Metal primers are easy to apply and seem to give positive results after APA and quite reliable bond strength with resin cements,[14,32,35] although doubts remain about hydrolytic stability.[14] The presence of adhesive monomer MDP in the mass of resin cement yields a stronger bond than other resin cements[28,127,128] and conventional cements,[9] or at least equivalent bond strength.[129] Among MDP resin cements, the role of the inorganic compounds are important in creating resistance to hydrolysis.[130] The adhesive potential to zirconia may be determined by other factors such as the particle size of fillers and viscosity.[131] The active parts of MDP react with the surface of zirconia, but these reactions are susceptible to instability after aging.[14,32,132] Without surface treatment, an MDP-metal primer (Alloy primer; Kuraray Co Ltd) appears to improve the chemical bonding with the resin cement,[32,133,134] while Yun et al[135] found a stronger bond with Alloy primer and V-primer (Sun), but only when preceded by APA with 90-μm alumina. MDP primers enhance the zirconia bonding values of acrylic resin cements.[108,136] A primer with MDP and a coupling agent for zirconia were mixed in various proportions and found to improve the bond to resin cements not containing MDP.[104] The combination of abrasion and a metal or ceramic activator also appears to improve the bond, but this bond strength is only maintained after aging in water in the case of the metal primer.[137] The use of new activators (zirconia primers) helps surface wetting by reducing the contact angle, but significantly less than fluorine plasma spraying.[112] In combination with silica coating, another universal primer (Monobond Plus; Ivoclar Vivadent AG) gave high bond strength values,[107,138] as did the Clearfil Ceramic Primer (Kuraray). A new zirconia primer containing organophosphate monomers and carboxylic monomers (Z-Prime Plus; Bisco Inc) was compatible with many resin cements and had a positive effect on bonding with resin cements after APA with 50-μm alumina[50,139] and even after an aging process.[38,107] The AZ Primer (Shofu Dental Corp) containing phosphonic acid monomers (6-MHPA) gave a better bond than other silane primers.[29] Metalprimer II (GC Corp) containing adhesive monomer thiophosphoric methacrylate (MEPS), gave a better and more aging resistant bond than ceramic primer (GC Corp).[137] At least 5 specialized formulations for zirconia bonding are available, and the number of available primers is increasing.[140] Another new specialized primer is Signum zirconia bond (Heraeus Kulzer GmbH), which appears even more effective.[141] Even without previous APA treatment, a novel universal primer containing both MDP and methacrylates promotes high bond strength.[142] Research indicates that each primer has a different optimal air-drying pressure.[143] Primers are now available for every different substrate (metals, ceramics, hard tissues).[144]

Bond strength after different treatments and aging

Predicting the behavior of materials after different aging tests to simulate intraoral use and recovering materials after use to study hardware failure are essential.[145] Bench aging procedures often differentiate among initially high bond strength values.[67] The hydrolytic action of water on adhesive surfaces and the inhibition reaction phenomena due to the presence of moisture are the main reasons for

Table 3. Selected shear bond strength tests without thermocycling

Author/Researcher Year	Zirconia Material	Surface treatment/ Resin Cement/ Primer	Best Method in MPa(SD)	TC/water Storage	No. of Specimens/Control Group
Akin 2011	Zirkonzahn, Zirkonzahn GmbH	Er:YAG laser/ NX3	3.2	7 d	30/group/YES
Atsu 2006	Cercon, DeguDent	CJ (30 µ)/Panavia F2.0, Clearfil primer	22.9 (3.1)	1 d	10/group/YES
Chen 2013	Cercon	APA 50 µ/Duolink/Z-prime plus	29.0 (6.3)	1 d	10/group/NO
Magne 2010	LAVA, 3M-ESPE	APA 50 µ/Z100 composite resin 3M/Z-prime plus	29.35 (5.11)	1 d	15/group/NO
Northdurft 2009	Digizon-A, AmanGirrbach	SC 30 µ/Bifix QM & silane	25.11 (4.86)	2 d	10/group/NO
Usumez 2013	Zirkonzahn, SRL	Nd:YAG laser/Clearfil Esthetic Cement	8.17 (1.9)	1 d	15/group/YES
Valentino 2012	Cercon, DeguDent	Glaze + HF + /Scotchbond Ceramic primer/Enforce	25.17 (8.37)	1 d	30/group/NO

APA, Airborne-particle abrasion; CJ, Co-jet; SC, silica coating.

Table 4. Selected tensile bond strength tests

Author/Researcher Year	Zirconia Material	Surface Treatment/ Resin Cement/ Primer	Best Method in MPa(SD)	TC/Water Storage	No. of Specimens/Control Group
Abouselib 2007	Cercon Base, DeguDent	SIE/Panavia F2.0 (Kuraray)	49.8-52.2	1 d-1 m	18/group/YES
Amaral 2014	Vita In Ceram YZ	APA (35 µ)/Scotchbond universal (3M ESPE)	33.8	1 d and TC 5-55 30 s dwell 2500c	15/group/YES
Attia 2011	E-max ZirCAD; Ivoclar Vivadent AG	SC 110 µ/Monobond plus/Multilink Automix (Ivoclar Viadent AG)	38.1 (6.2)-45.2 (4.7)	3-150 d with TC 37<ts>5000c	16/group/NO
Palacios 2006	Procera AllZircon; Nobel Biocare	APA 50 µ/Panavia F2.0 (Kuraray)	6.9 (2.9)	5000c TC	12/group/NO
Wolfart 2007	Cercon; DeguDent	APA 50 µ/Panavia 2.0 (Kuraray)	39.2-45.0	3 d/150 d and 37500c TC	20/group/NO

APA, airborne-particle abrasion.

up to 50% reductions from the baseline bond strength.[71] Water thermocycling causes repeated thermal expansion and contraction of the materials used, which causes fatigue at the interphase and therefore a reduction in bond values.[146] The most common tests applied involve long-term storage in an aqueous environment and hydrothermal recycling,[53] which significantly reduce the initial bond strength values in tensile, shear, or push-out tests.[1,31,127,130,137] Most experiments contain a separate analysis of the results before and after the aging process, sometimes with dramatic reverses in bond strength values.[43,147] De Castro et al[148] observed that the aging process did not significantly affect the bond strength in tensile testing, regardless of the type of luting agent. With MDP resin cements and surface pretreatment, no significant changes were found after hydrothermal recycling.[56,149] In contrast, in polished zirconia surfaces, many spontaneous detachments occurred after an aging process, despite the influence of activators.[41]

Surface contamination avoidance techniques

The bonding surfaces of ceramics often become contaminated by saliva, blood, silicone pastes, residual gypsum, and rubber gloves.[150] Various methods have been tested for removing the surface layer, including organic solvents, acids, abrasive grained alumina, washing with water, and ultrasonics.[151,152] The affinity of zirconia with the phosphate group is known, so when phosphoric acid is applied to a zirconia surface, positions that could be covered by adhesive phosphate monomers become inactive.[153] Therefore, the most recommended method is cleaning with a mixture of zirconia powder and sodium hydroxide (Ivoclean; Ivoclar Vivadent AG). The use of an ethyl cellulose protective lacquer seems to inhibit the negative results of intraoral contamination on APA surfaces.[154]

RESULTS

Twenty-three of the most relevant experimental articles, in our opinion, were selected and are shown in Tables 2-4. Articles with a minimum sample size of 10 specimens were included. The evidence shows that the resin cements with the highest long-term bond strength contain MDP or use MDP-primers. Also, in most protocols, APA and TBC had the best performance. Occasionally, alternative methods (coatings, lasers, SIE) also showed high bond strength values.

DISCUSSION

The plethora of techniques that have already been tested reinforces the difficulty of obtaining a reliable long-term bond in vitro. Two basic experimental designs, shear and tensile tests, are used on both the micro and macro scale. Shear tests involve simpler experimental apparatus and protocols than tensile tests, and loading direction is of little importance and has almost no impact on the results.[155] However, shear tests are criticized for the nonhomogeneous distribution of stresses in the adhesive interface, which can lead to an overestimation or misinterpretation of results.[156,157] Tensile tests evaluate real adhesion bond strength more reliably, although most researchers follow the shear test design (macroshear).[157]

Increased surface roughness provides a more extensive area for adhesion, but accurate measurement of roughness is a complex process, and more parameters should be investigated for a more reliable description.[158] Also, the role of the adhesive monomers and silanes is important.[43] The agonistic or antagonistic action of chemical compounds and the exact contribution of factors affecting the adhesion processes in the final result could be assessed further with more specialized experimental investigation. Aging procedures often reduce the initially high bond strength values and simulate the operating conditions.[71] It is important to compare the results of both before and after the aging process and to evaluate bond strength resistance to hydrolysis and constant temperature variations.[43,147]

The bond strength values of zirconia and dental cements vary greatly. Different zirconia materials, the type of experimental set-up (tensile, shear, or push-out), the size of specimens, the variety of materials and processing techniques, as well as specimen storage conditions are the main variables that cause difficulty in the direct comparison of results.[43,157] Even without any surface treatment the bond strengths are clearly very low, and, after aging, nonexistent. The use of adhesive monomers gives satisfactory results, but aging tests reveal long-term instability. Coating techniques also seem promising but are usually complicated, and the stability of a coating to a zirconia substrate has not been thoroughly investigated. Mechanical treatment, and especially APA, is considered as "gold standard" and almost invariably increases bond strength. Laser treatment is still controversial. On the side of the mechanical pretreatment of the surface, there is also wide scope for research. The change in surface texture with pioneering subtractive methods (laser, APA, diamond rotary instruments) or with various coatings may also alter the mechanical retention of a resin material. The field of research for the development of a reliable protocol for optimum zirconia bonding is still open.

Clinical implications or recommendations are difficult to give, because bond strength tests are only relative, indicative of the superiority of one method over another. Moreover, laboratory test results need to be confirmed by clinical studies before a certain cementing protocol is given. There is still no universal surface treatment for clinically sufficient bonding of zirconia ceramics.

Further improvement of the adhesive capacity and compatibility of resin cements to zirconia ceramics will be achieved by isolating the factors that contribute positively to the bond. The data concerning chemical bonding (adhesive monomers, silanes) must be analyzed individually to determine their contribution to the adhesive strength. The next area of research will be the synthesis of new resin materials or specialized primers with different proportions of adhesive monomers to ensure the maximum bond to zirconia. In parallel, RCTs are necessary to confirm laboratory measurements and draw conclusions under oral conditions to support or refute some methods and adopt, eventually, a specific protocol for the bonding of zirconia restorations.

CONCLUSIONS

Based on the findings of this systematic review, the following conclusions were drawn:

1. APA is a reference method included in most research protocols.
2. TBC enhances bonding capacity, especially when silanes are applied.
3. Adhesive monomers are necessary for chemical bonding.
4. Surface contamination and aging have negative effects on adhesion to zirconia.
5. Laboratory studies have limits. The role of aging is important to most research protocols but should be confirmed by clinical trials.

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