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Adhesion to Zirconium Dioxide Used for Dental Reconstructions: Surface Conditioning Concepts, Challenges, and Future Prospects

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Abstract Yttrium-stabilized zirconium dioxide, a commonly used material in conjunction with CAD/CAM technologies in dentistry, is an oxide ceramic that does not comprise the silicon dioxide (SiO_2) phase in its microstructure. Since it is challenging to create durable adhesion between resin cements and this kind of non-etchable ceramic, efforts have been made to develop innovative surface conditioning methods over the years. While some chemical methods based on using adhesion promoters only did not perform stable adhesion, others utilizing physico-chemical conditioning methods provided better adhesion where the latter is also being questioned on impairing mechanical stability of zirconium dioxide (ZrO_2) due to $t \rightarrow m$ phase transformation. This review will highlight current surface conditioning concepts to achieve best adhesion to zirconium dioxide and challenges related to conditioning methods or resin-based luting cements, and contemplate on future prospects.

Keywords Adhesion · Resin-bonded fixed dental prosthesis · Resin cements · Surface conditioning · Y-TZP · Zirconium dioxide

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Introduction

With advances in adhesive dentistry over the last few decades, and especially after the introduction of CAD/CAM technology, many clinical cases restored with metal-ceramic fixed dental prostheses (FDP) are currently being solved with metal-free materials, one of which is yttrium-stabilized tetragonal zirconia polycrystal (hereon zirconia).

Zirconia as a Material

Among numerous other ceramic options, zirconia drew considerable attention of the scientists and clinicians in the dental field due to its favorable mechanical properties [1, 2]. This biocompatible, chemically bioinert ceramic has potential for versatile applications in dentistry other than FDPs such as root posts, orthodontics brackets, implants, or implant abutments.

The microstructure of zirconia consists of about 2.5 to 3.5 % yttrium oxide (Y_2O_3) and is formed mostly by particles of micrometric ZrO_2 after sintering from a metastable tetragonal structure at room temperature [3, 4]. Zirconia has a standard density of 6 g/cm^3, while its theoretical density is 6.51 g/cm^3. The closer these two values are, the smaller the interparticle space and, thereby, the greater the strength and the smoother the surface of the material [5].

Zirconia and Transformation Change

As a function of temperature alteration, pure zirconia exhibits three polymorphic phases, also known as allotropic or crystalline phases. While at temperatures up to 1170 °C the material presents a monoclinic phase, tetragonal structure can be observed at intermediate temperatures (1170–2370 °C) and finally the material exhibits a stable cubic structure at high temperatures (>2370 °C) [3, 6]. Among all these three phases, the monoclinic phase has inferior mechanical properties, which actually helps reduce the cohesion between particles but at the same time diminish the final density of the material. In order to be able to use zirconia as a framework material for FDPs, it is essential to have stabilized tetragonal or cubic phases at room temperature. For better control of the phase transformation, oxides (CaO, MgO) and rare earths (CeO_2, Y_2O_3) are used as stabilizers preventing phase transformations that may occur during veneering procedures [7].

According to the ISO assigned for biomedical applications, Y_2O_3 is extracted from a xenotime mineral and it is the most commonly used stabilizer for such applications [8]. Unfortunately, when subjected to pressure or any kind of impact, the stabilized zirconia can undergo tetragonal metastable to monoclinic ($t \rightarrow m$) transformation [1, 3, 4]. This type of transformation is accompanied by a volume increase of 3 to 5 % that tends to induce compressive stress in the transformed region that in turn prevents crack propagation [3]. Toughening by transformation is strongly dictated by the presence of defects on material, size of ZrO_2 grains, type and amount of stabilizers, and radiant of temperature [9•]. Small grains are essentially more prone to transformation than larger grains, but there is a critical grain size, below ≈0.2 μm, where the transformation does not occur. However, above that grain size, transformation occurs spontaneously [10]. Moreover, while some grains undergo transformation at room temperature, others require an increased temperature for this process mainly due to their smaller size [10, 11].

Surface Conditioning Methods for Zirconia

Hydrofluoric acid (HF) etching followed by the application of silane coupling agents is a well-established conditioning method and delivers durable adhesion to glassy matrix ceramics used for dental applications. Unfortunately, this method does not provide adequate adhesion to zirconia since it does not contain a silica phase. Achieving durable resin cement adhesion to zirconia becomes particularly crucial to increase the clinical indication of surface-retained resin-bonded FDPS where the retention solely relies on durable adhesion of the tooth-resin-zirconia complex. While some surface conditioning methods facilitate resin-ceramic bonding micromechanically using airborne particle abrasion with alumina particles or alumina, other methods are based on physico-chemical activation of the surface using silica-coated alumina particles or silica particles alone followed by silanization [9•, 12••].

Alternative surface modification methods such as selective infiltration etching (SIE) [13, 14], glaze ceramic coating [15], chemical plasma vapor deposition using hexamethyldisiloxane or chlorosilane gas [16], erbium-doped yttrium aluminum garnet (Er:YAG) laser application [15, 17], and physical vapor deposition using a magnetron sputtering technique (RMS) [18, 19] have also been tried in an attempt to improve adhesion to zirconia. In the SIE method, low temperature fused glass ceramic is applied to the selected surface that is then etched with 5 % HF solution. Etching process establishes porosities between the grains that are then filled with low-viscosity resin cement [14]. Despite the fact that this method provides initially favorable adhesion, hydrolytic stability after aging tends to reduce the achieved results [13]. On the other hand, the plasma spraying method, using hexamethyldisiloxane, is a rapid process that can be accomplished at low temperatures. In this method, a reactor generates an ionized gas deposited on the zirconia surface where the chemical configuration and film thickness can be controlled during application. Nevertheless, the adhesion mechanism is not yet clear and, in fact, the working mechanism is postulated to work with covalent bonds between adhesive luting cement and zirconia rather than the chemical reaction achieved by the plasma coating [16]. Similarly, the RMS method is based on deposition of high-energy particles on solid surfaces creating free radicals. The released particles scatter through the plasma of the inert gas at low pressure, depositing a uniform and controlled film and activating the substrate for improved adhesion without compromising the mechanical properties of the substrate [18, 19].

One other method known as Er:YAG laser ablation uses laser irradiation that creates a rough zirconia surface [17, 20]. Yet, studies have shown that laser irradiation is not as effective as airborne particle abrasion methods in improving adhesion of resin cements to zirconia [21]. Other coating methods using micropearls of low-fusing ceramic or vapor deposition of silicon tetrachloride ($SiCl_4$) have shown worthy results of adhesion [22], but again, glass deposition was found critical to interfere with the internal adaptation of the reconstruction [23, 24].

Achieving a clean substrate surface is essential for durable adhesion to all materials. In that respect, air abrasion eliminates organic contaminants such as cement, plaster, and saliva debris from the ceramic surface after the clinical and laboratory steps [25••, 26•, 27] that increases the wetting kinetics of adhesives [28–31]. Moreover, due to surface roughness, an increase in surface area of approximately 80 % can be attained that expands bonding sites available to react with an adhesive promoter [3]. In dentistry, air-abrasion systems are practiced using different particle types and sizes ranging from 30 to 250 μm [3, 12••] where particles of 110–250 μm are typically used in dental laboratories and 30–50 μm particles at chairside [3, 12••, 19, 32–35].

The most commonly used particle types are alumina (Al_2O_3) or alumina-coated silica (SiO_2). When alumina particles are coated with silica through the sol-gel method, a silica layer is deposited on the zirconia, which allows interaction with the subsequently applied silane coupling agents, forming hydrolytically more stable –Si–O–Si–O– bonds compared to

–Al–O–Si– [36]. The silane coupling agent with its bifunctional characteristics is capable of reacting with silicon dioxide (SiO_2) deposited on the zirconia surface, and copolymerizes with the organic matrix of the resin luting cement [10, 36]. This technique known as tribochemical silica coating [32] demonstrated better adhesion results between resin luting cements and zirconia as opposed to conditioning the surface with conventional Al_2O_3 particles [12••, 16, 25••, 33]. It has to be emphasized that the effectiveness of air-abrasion methods depends highly on the deposition parameters such as type and size of the particles, pressure, angle and distance of the nozzle in relation to the substrate surface, and duration [37]. Different particle sizes ranging from 25 to 250 μm, pressure from 0.5 to 7 bar, and duration of 5 to 20 s were employed in previous studies which make it difficult to make direct comparisons of the results [12••]. Fortunately, recent meta-analysis of the published data verified the efficacy of air-abrasion concepts for improved adhesion to zirconia [12••, 25••].

Concerns on Air Abrasion

To date, controversial opinions are present regarding the harmful effects of particle deposition methods on zirconia [38, 39, 40••, 41, 42], in that momentum of particles on zirconia yields to local lattice distortions or creates a new phase by ferroelastic domain switching. In fact, the impact of particle deposition methods on zirconia is dependent on parameters such as particle morphology, pressure, and duration [37] effecting a residual compressive stress layer that stimulates phase transformation ($t \rightarrow m$) and creates transformed zone depth (TZD) [9•, 40••]. TZD typically ranges from 0.59 to 1.6 μm, and interestingly, with the good combination of deposition parameters, this zone may even increase the mechanical strength of zirconia [38, 40••, 41, 42]. ISO standard 13356 suggests 25 % as the maximum acceptable amount of monoclinic phase [8]. In fact, the highest amount reported so far even in the harshest conditions barely exceeded this limit. Thus, delicate application of air-abrasion protocols, using a smaller particle size of 50 μm or less, as short as possible (approximately 20 s/cm^2) between 0.5 and 2.5 bar and with controlled nozzle distance of approximately 10 mm, is among currently accepted conditioning parameters [9•, 35, 37]. The increase in pressure during air-abrasion procedures can produce even deeper surface morphology, hindering the wettability and flow of resin luting cement into the irregularities [39, 42].

Resin Luting Cements

The choice of resin cements also plays a significant role in adhesion to zirconia. Phosphate ester monomer-containing adhesion promoters with polar functional groups such as 10-methacryloyloxydecyl dihydrogenphosphate (10-MDP) [12••, 43, 44] could make covalent bonds with the hydroxyl groups on the zirconia surface. Currently, alumina or silica coating followed by the application of MDP-containing silane coupling agent and MDP-based resin cement appears to be more resistant to aging [12••, 45] compared to methacrylate-based conventional resin luting cements [12••, 25••, 33, 46].

While concerns exist about the potential damage by air-abrasion protocols on zirconia, some manufacturers started to promote chemical activation of the surface with self-adhesive cements. When conventional resin cements are used, conditioning zirconia surface is mandatory. In contrast, self-adhesive resin cements do not require physical and/or chemical conditioning of neither zirconia nor dental tissues, saving clinical time, which also do not compromise the strength of zirconia. This type of self-adhesive cements show great variation in their chemical compositions in that while some contain phosphoric acid esters, MDP, bis-HEMA-phosphate, glycerolphosphate dimethacrylate, and 4-META, others contain bis-GMA alone or in combination with TEG-DMA. Unfortunately, low degree of conversion of such acidic monomers, less hydrolytic stability, and low diffusion level into dentin are limitations of self-adhesive cements [47, 48].

On Aging of Adhesive Interfaces and Zirconia Itself

In adhesion studies, the bonded joints are subjected to different aging conditions to estimate the long-term clinical behavior [49••, 50]. While water storage mimics aging due to water uptake and hydrolytic degradation, thermocycling characterizes hydrothermal aging where the latter significantly reduces adhesion [51]. Aging conditions show big variation, making comparisons between studies difficult. Some standardization on the aging protocol seems to be crucial but generally contingent on the specimen dimensions for each in vitro test method; water storage may result in hydrolysis of the adhesive joint similar to thermocycling. In a systematic review of parameters in dentin bonding studies, both thermocycling and long-term water storage did not show high sensitivity but water storage presented a greater bond-degrading effect [52]. Future studies on adhesion tests should consider the degrading effect of water storage versus thermocycling.

Similar to adhesion-related studies, fatigue studies on zirconia also present huge scatter of fatigue parameters in aging this material. In the dental literature, cycling loading of minimum 10,000 cycles at 1 Hz between 30 and 300 N and maximum 1,200,000 cycles at 50 N at 1.3 Hz are practiced [3]. The use of dynamic and cyclic tests after air abrasion with 50 μm alumina or alumina particles coated with silica resulted in significant reduction in surface flaws and could even improve fatigue resistance of zirconia [53].

Concluding Remarks

Retention loss of zirconia-based crowns or FDPs is a rare event according to the clinical studies [54, 55, 56••, 57, 58]. Only in the case of minimally invasive resin-bonded FDPs made of zirconia is durable adhesion essential. Since many confounding factors are present in clinical trials, in vitro studies will remain to serve for ranking materials and identify the best performing ones in worst-case scenarios prior to such trials. At the moment, MDP-based adhesive promoters and resin cements seem to deliver reliable adhesion to zirconia. In addition, air abrasion increases micromechanical retention, but moderately rough surface that does not yield to monoclinic phase formation requires meticulous air-abrasion procedures. Clinicians should also note that durable adhesion of zirconia FDPs to enamel and dentin requires adequate surface conditioning of the two substrates, which may be based on several steps, and the choice of resin cement that adheres to both substrates [59]. Future developments in surface conditioning methods aim for reducing or completely eliminating these multistep procedures and achieving a surface on zirconia that delivers adhesion similar to glassy matrix ceramics or polymeric materials. This will mean either further development of resin cements that do not suffer from aging or new conditioning methods that have more potential for chairside applications.

Compliance with Ethics Guidelines

Conflict of Interest The authors declare that they have no conflict of interest.

Human and Animal Rights and Informed Consent This article does not contain any studies with human or animal subjects performed by any of the authors.

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