Ultrastructure and bonding properties of tribochemical silica-coated zirconia

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Tribochemical silica coating (TSC) is commonly used to pretreat zirconia surfaces prior to luting. Although many studies demonstrate an adhesion-promoting effect of TSC on zirconia, its actual interaction mechanism has not been fully elucidated. We therefore characterized the ultrastructure of TSC-treated zirconia and tested shear-bond strength. STEM/EDS disclosed a micro-roughened zirconia surface partially covered with fused Al and Si, while residual unfused silica particles could also still be detected. TSC-treated zirconia having received the solely silane primer exhibited a significantly lower shear-bond strength than zirconia on which the combined 10-MDP/silane primer was applied. SEM fracture analysis revealed residual silica particles on both the zirconia and cement sides. Correlative ultrastructural and chemical surface characterization revealed that TSC deposited an inhomogeneous silica layer on the zirconia surface, which explains why the solely silane coupling agent was less effective than the combined 10-MDP/silane ceramic primer for bonding to zirconia pretreated by TSC.

Keywords: Zirconia, Tribochemical silica coating, Silane coupling agents, Scanning electron microscopy, Transmission electron microscopy

INTRODUCTION

The increasing demand for aesthetics has promoted the clinical use of tooth-colored ceramic restorations. Beyond the possibility to match the ceramic to the patient's teeth regarding color and overall natural appearance, metal-free ceramics are also advantageous to patients with an allergy to metal. These metal-free ceramics must have high mechanical properties, such as strength, toughness and fatigue resistance, so to be used for molar crowns and bridges. For this reason, many different types of ceramics, such as aluminosilicate glass-ceramics, lithium (di)silicate glass-ceramics, densely sintered alumina and zirconia have been developed and routinely used in clinic[1]. Among them, zirconia exhibits the most promising properties to combine strength with aesthetics for diverse dental applications, such as all-ceramic partial and full crowns and bridges, and even implants and implant abutments[2], this in particular now bilayered veneered zirconia is evolving to translucent full-contour graded zirconia.

For successful clinical application, ceramic materials are best adhered to the remaining tooth structure *via* cements that bond both to enamel/dentin as to the restoration substrate. In addition to mechanical properties, the bonding receptiveness and adhesive

procedures of/for ceramics should thus be considered as well. Basically, strong and durable bonding depends on both (micro-)mechanical and chemical bonding, this apart of good wetting properties of the adhesive/cement as well. Sandblasting with aluminum-oxide (Al_2O_3) particles can be used to enhance micro-mechanical interlocking, as it cleans and micro-roughens the restoration surface to the direct benefit of micro-retention[3]. Doing so, a high 'immediate' bond strength to zirconia can be reached, which however appeared sensitive to aging despite the sandblasting pre-treatment[4]. In other words, solely sandblasting was shown insufficient to reach durable bonding to zirconia.

Silane coupling agents are often used to promote adhesion to ceramic materials. Silanol groups existing at the silica-based ceramic surface are receptive for silane coupling. However, for non-silica-based ceramics, such as zirconia, the adhesion performance using silane coupling agents is not satisfactory[5].

Another way to improve bond strength to ceramics involves the application of tribochemical silica coating (TSC), an active type of sandblasting, this for instance using the Rocatec or Cojet systems (3M, Seefeld, Germany). These systems generate a fresh silica-rich ceramic surface, thereby promoting silane coupling onto the ceramic surface. Although the surface topography and roughness of zirconia pretreated by TSC has been analyzed[6-9], the actual interfacial ultra-structure at

TSC-treated zirconia has not (yet) been completely elucidated.

In this study, the ultrastructure of zirconia surfaces treated by TSC using 30 µm Rocatec Sand powder and Rocatec system was investigated using high-resolution scanning electron microscopy (SEM) and transmission electron microscopy (TEM) in combination with chemical element analysis using energy dispersive spectroscopy (EDS). These microscopic observations were correlated with shear bond-strength measurements. The aim of this study was to test our hypothesis that TSC-treated zirconia surfaces are completely coated by a fused silica layer.

MATERIALS AND METHODS

Preparation of tribochemical sand specimens for SEM
Rocatec Soft sand consisting of 30-µm diameter silica-coated Al_2O_3 particles were embedded in epoxy resin (G2 epoxy glue, Gatan, Pleasanton, CA, USA). The embedded specimens were next prepared for cross-sectional SEM using argon-ion milling (SM-090101 Cross Section Polisher, JEOL, Tokyo, Japan). Subsequently, a thin layer of osmium was vaporized onto the specimen surface using a Neo Osmium Coater (Meiwa Fosis, Tokyo, Japan) prior to SEM examination (JSM6701F, JEOL).

Preparation of TSC-treated zirconia specimens for SEM and TEM
Three 3-mol% sintered Y_2O_3-stabilized zirconia plates ($10\times10\times3$ mm^3) were obtained from Tosoh (Tokyo, Japan). The plates were tribochemically silica-coated with silica-modified aluminum oxide (Rocatec Soft sand, 3M) for 13 s/100 mm^2 at a pressure of 0.28 MPa and at a 10-mm distance, as per manufacturer's instructions. The remnants of sand particles were blown away by a strong air flow. The zirconia specimens subjected to TSC are hereafter called 'TSC-treated zirconia'. The surface was next treated by silane coupling agent (ESPE Sil, 3M) for 30 s. One of these specimens was prepared for SEM by vaporizing a thin layer of osmium onto its surface using the Neo Osmium Coater (Meiwa Fosis). The other two specimens were prepared for TEM/STEM by coating their surface with G2 epoxy glue (Gatan), followed by cross-sectioning using argon-ion milling with an ion slicer (EM-09100IS, JEOL).

TEM/STEM examination and EDS analysis of TSC-treated zirconia
The TEM and STEM studies were performed using a 300-kV TEM (JEM-3010, JEOL) and a 200-kV TEM/STEM (JEM-2100F, JEOL). The JEM-2100F was equipped with a probe-forming Cs corrector (CEOS, Heidelberg, Germany), STEM bright-field (BF) and annular dark-field (ADF) detectors, and EDS (JED-2300T, JEOL). The probe-forming Cs corrector enabled sub-Å resolution STEM imaging with a minimum probe size of 0.09 nm.

Shear bond-strength tests
The different materials used for the shear bond-strength test are listed in Table 1. Specimen preparation is detailed in Fig. 1. The surface of twenty zirconia plates ($10\times10\times3$ mm^3; Tosoh) were polished using 15-µm diamond lapping film, prior to being TCS-treated with 30-µm Rocatec Soft sand using the Rocatec Plus sandblaster at a blast pressure of 0.28 MPa for 15 s. The distance between the blast nozzle and zirconia surface was set to 10 mm. The surfaces were then cleaned with a soft air blow for 5 s. On half of specimens, ESPE Sil was applied on the TSC-treated zirconia surface using a disposable micro-brush. After applying ESPE Sil, the surfaces were allowed to dry in air for 5 min (according to the manufacturer's instructions). The other half of the TSC-treated zirconia specimens were treated using the combined 10-MDP/silane-containing Clearfil Ceramic Primer (Kuraray Noritake Dental, Tokyo, Japan), followed by gently air-blowing after 5 s (according to the manufacturer's instructions).

In addition, the surfaces of twenty zirconia cylinders (3.4-mm diameter, 3-mm thickness; Tosoh) were sandblasted with 50-µm Al_2O_3 particles using a laboratory sandblaster (Hi-Blaster III, Shofu, Kyoto, Japan) with a blast pressure of 0.4 MPa for 10 s at a distance of 10 mm[10]. The sandblasted surfaces were next treated

Table 1   Composition of the materials used for the shear bond-strength test

Material	Manufacturer	Composition
Clearfil Ceramic Primer	Kuraray Noritake Dental, Tokyo, Japan	3-MPS, 10-MDP, ethanol
ESPE Sil	3M ESPE, Seefeld, Germany	3-MPS, ethanol
Clearfil Esthetic Cement	Kuraray Noritake Dental	Paste A: TEGDMA, methacrylate monomers, silanated glass filler, colloidal silica Paste B: Bis-GMA, TEGDMA, methacrylate monomers, silanated glass filler, silanated silica, colloidal silica, benzoyl peroxide, CQ, pigments

3-MPS: 3-methacryloxypropyltrimethoxysilane, 10-MDP: 10-methacryloxydecyl dihydrogen phosphate, Bis-GMA: bis-phenol-A-diglycidyl methacrylate, CQ: camphoquinone, TEGDMA: triethyleneglycol dimethacrylate.
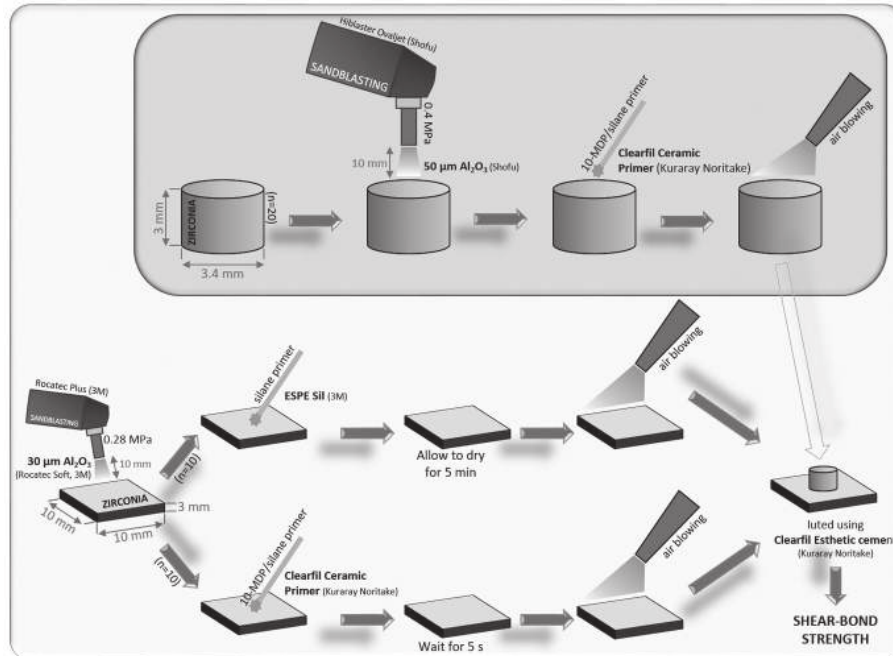
Fig. 1   Schematic detailing specimen preparation for the shear bond-strength test.

with Clearfil Ceramic Primer, upon which the zirconia cylinders were luted onto the zirconia plates with a resin-based luting cement (Clearfil Esthetic Cement, Kuraray Noritake Dental) using finger pressure (corresponding to a pressure of about 2.2 MPa measured by mechanical force gauge (PS-100N, Imada, Aichi, Japan, n=10)[10]. These specimens were polymerized using a light-curing unit (average of 2,800 mW/cm^2 light irradiance; G-Light Prima II Plus lamp, GC, Tokyo, Japan) for 1 min. After polymerization, the specimens were stored in distilled water at 37°C for 24 h.

The specimens were next mounted into a testing machine (Instron 5565, Instron, Canton, MA, USA), upon which the specimens were subjected to shear stress at a cross-head speed of 0.5 mm/min. Upon failure, the fracture mode was determined under 4× magnification using a light microscope (SMZ-10, Nikon, Tokyo, Japan). Selected fractured surfaces were examined by SEM as well.

The shear bond-strength data were statistically compared using one-way analysis of variance (ANOVA) with a p-value below 0.05 being considered as statistically significant.

RESULTS

SEM of tribochemical sand specimens
SEM of Rocatec Soft sand revealed irregular particles in size and shape (Fig. 2a). High magnification demonstrated the presence of small silica particles around each alumina particle (Fig. 2b). The diameter of these silica particles was around 50 nm; they were densely packed (Fig. 2c). Cross-section SEM of Rocatec

Soft sand embedded in epoxy resin disclosed that main outer part of the large alumina particles was covered with the small silica particles (Figs. 2d, f). High-magnification cross-sections revealed the presence of agglomerated silica particles (Fig. 2f).

SEM of TSC-treated zirconia specimens
TSC-treated zirconia revealed a very rough surface exhibiting plate-like layers (Fig. 3). High-magnification SEM image disclosed the deposition of 50-nm spherical particles partially covering the zirconia surface (Fig. 3c).

Cross-section TEM and STEM-EDS mapping of TSC-treated zirconia
High-resolution (HR) TEM confirmed the roughness of the TSC-treated zirconia surface (Fig. 4a). Two almost triangular-shaped features were observed in Figs. 4a and b (appearing medium-grey; marked by the open arrows in Fig. 4b). High-angle annular dark-field (HAADF) imaging also displayed this structure clearly, as did the EDS mapping for silicon (Si) and oxygen (O) as well (Figs. 5a, b). These triangular-shaped structures appeared homogeneous, indicating a solidified melted coating. Other parts of the zirconia surface clearly did not exhibit this type of structures (Figs. 4c, d, interrupted open arrows).

EDS mapping furthermore confirmed that the small particles observed in Figs. 4a and b were composed of silicon oxide. EDS mapping of aluminum (Al) exhibited another triangular-shaped feature, which can be seen on the O mapping as well.
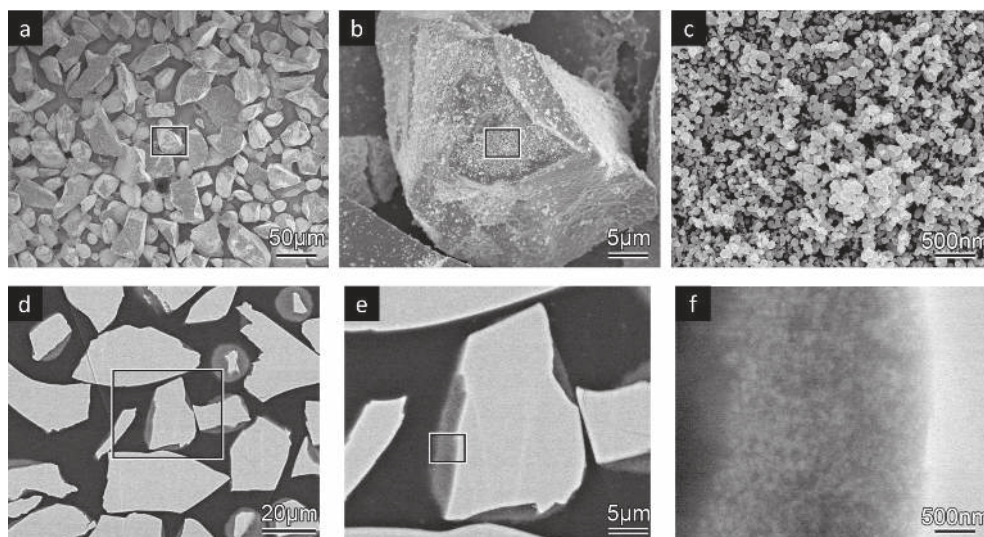
Fig. 2   SEM photomicrographs of Rocatec Soft (3M) particles.
(a–c) Surface images; (d–f) cross-section images.
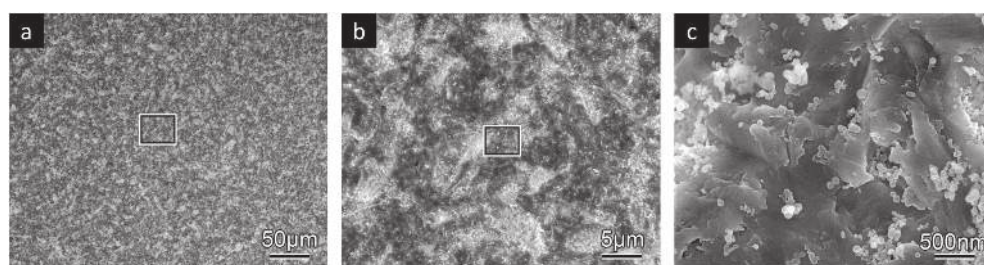

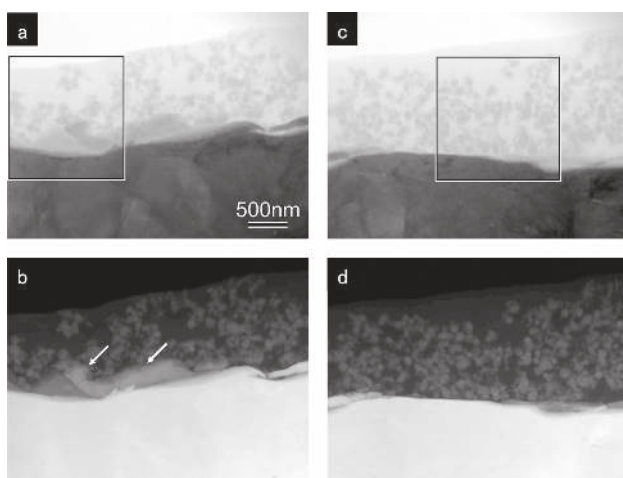Fig. 3   SEM photomicrographs of the TSC-treated zirconia surface.

Fig. 4   Cross-section HRTEM photomicrographs of TSC-treated zirconia.
(a, c) Bright-field images; (b, d) Dark-field images.

Shear bond-strength tests
The mean shear-bond strength (in MPa) along with the corresponding standard deviation is presented for both types of specimens in Fig. 6. The specimens treated with the solely silane-containing ceramic primer ESPE Sil revealed a significantly lower bond strength than the specimens treated with the combined 10-MDP/silane-containing primer Clearfil Ceramic Primer. All specimens broke either at the cement-zirconia plate interface, within the cement, or following a mixed failure mode. No specimen fractured at the interface between cement and zirconia cylinder.

SEM of fractured specimens subjected to TSC and ESPE Sil revealed the presence of Rocatec Soft sand particles on both the zirconia and cement sides (Fig. 7).

DISCUSSION

Rocatec Soft sand is made of 30-µm diameter Al_2O_3 particles coated with a thin layer of silica particles[11]. Although many studies investigated zirconia surfaces treated with Rocatec Soft or Cojet sand[6,9,11-20], the ultrastructure of Rocatec Soft/Cojet sand has not been characterized before. In this study, our observations showed that Rocatec Soft sand consisted of sharp Al_2O_3 particles of irregular size, having a diameter ranging from 10–70 µm, while being covered with silica particles
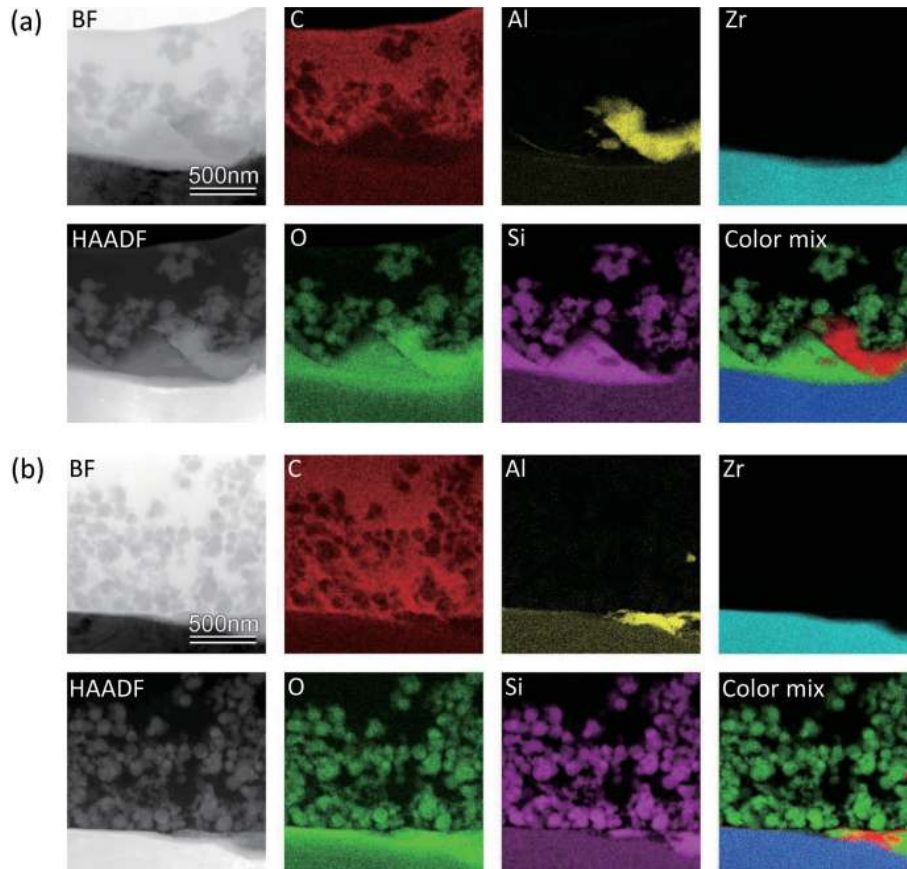
Fig. 5 Bright-field (BF) and high-angle annular dark field (HAADF) STEM photomicrographs along with EDS mapping images of TSC-treated zirconia.
EDS mapping revealed the distribution of carbon (C), aluminum (Al), zirconium (Zr), oxygen (O) and silicon (Si), respectively. The 'color mix' image shows all detected elements in one image. (a) Area indicated in Fig. 4a; (b) area indicated in Fig. 4c.
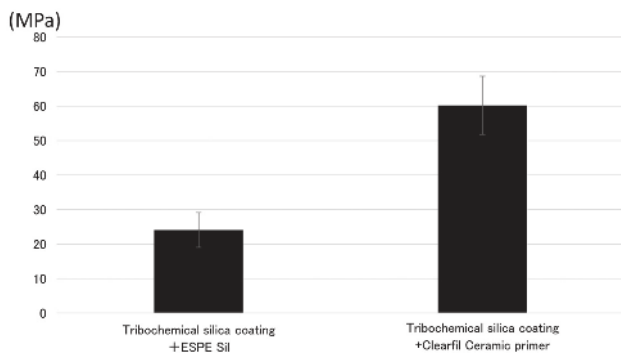

Fig. 6 Mean shear-bond strength (in MPa) and the corresponding standard deviation for TSC-treated zirconia additionally exposed to the solely silane-containing ceramic primer ESPE Sil (3M) or the combined 10-MDP/silane-containing primer Clearfil Ceramic Primer (Kuraray Noritake Dental).
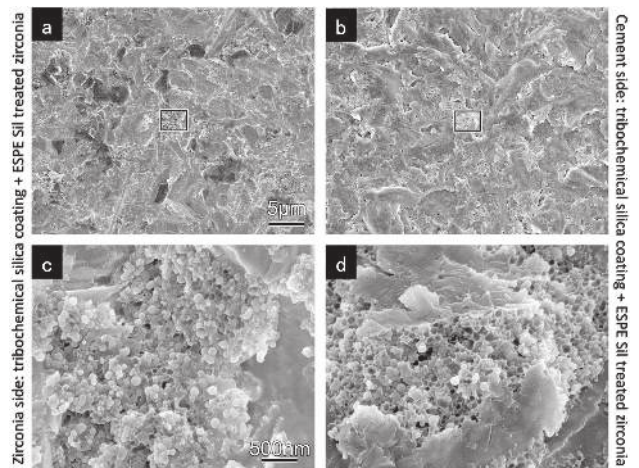

Fig. 7 SEM of fractured surfaces revealing the zirconia (a, c) and cement side (b, d).

with a diameter of around 50 nm or with a thicker SiO_2-particle layer. Rocatec Soft sand is used for so-called 'tribochemical silica-coating' (TSC), also known as

'silicatization'[21]. The deposited coating layer is suggested to be chemically bonded to zirconia. Furthermore, it has been described to be reactive with organosilane coupling

agents, contained in classical solely silane-containing ceramic primers, such as ESPE Sil used in this study, or in the newer so-called 'universal' ceramic/restoration primers, in which silane is mixed with other functional monomers, such as 10-MDP as in Clearfil Ceramic Primer. Coupling the silane functional monomer to the zirconia surface enables the composite cement to co-polymerize with the silane monomer's methacrylate end. To ensure good adhesion of cement to zirconia through silane coupling, TSC should ideally coat the zirconia surface completely with silica. However, we observed that some areas of the zirconia surface remained uncoated, this depite the manufacturer's instructions were strictly followed. Our hypothesis that TSC-treated zirconia surfaces are completely coated by a fused silica layer, was thus rejected.

Hallmann *et al.* found that the alumina ratio on zirconia surfaces increased with increasing blasting pressures[22]. A higher blasting pressure might thus be needed to form a more homogeneous silica layer. We found that a blasting pressure of 0.28 MPa, being the pressure prescribed by the manufacturer, appeared insufficient to achieve a complete tribochemical process. On the other hand, a higher blasting pressure may also induce phase transformation of zirconia and so damage the zirconia surface[22].

Our observation that some silica particles were not in contact with the zirconia surface and/or did not fuse with other particles, confirmed that the tribochemical reaction was not complete. Another SEM observation disclosed many loose silica particles on TSC-treated zirconia surfaces[23]; one study revealed that ultrasonic cleaning could remove the silica particles that appeared rather loosely deposited on zirconia by TSC[12].

Our understanding of the tribochemical process, based on the results of this study, is schematically represented in Fig. 8. When silica-coated alumina particles hit the zirconia surface, their kinetic energy is partially converted into thermal energy, causing a local increase in temperature (Figs. 8a and b). Some of the silica particles therefore melt and adhere to the zirconia surface (Fig. 8b). Simultaneously, some alumina particles fracture, causing fragments to be attached onto the zirconia surface. A lot of silica particles did however not melt but were deposited as single particles onto the surface and could remain despite air blowing (Fig. 8c).

TSC has been shown to promote the surface receptiveness of zirconia for chemical interaction with silane coupling agents[19]. On the other hand, any potentially negative effect of unfused silica particles remaining at the zirconia surface on the bond strength has not been reported before. The fracture analysis in this study revealed the presence of residual silica particles on both fracture sides. This may indicate that non-melted residual silica particles may have inhibited adequate bonding of the cement to zirconia, thereby to have caused the interface to preferentially fracture at that particularly silica particle-rich zone at the cement-restoration interface.
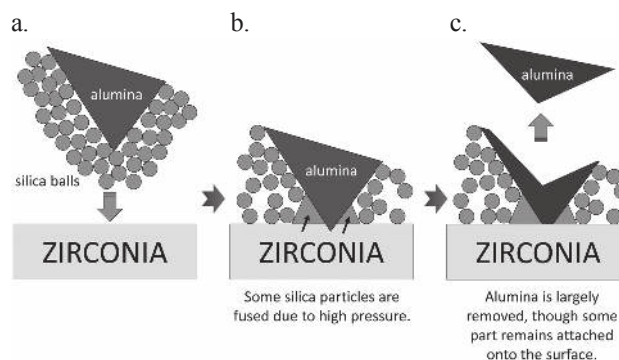
Nishigawa *et al.* detected the deposition of Al on

Fig. 8 Schematic explaining the TSC process onto zirconia.

zirconia surfaces when treated with 110-µm Rocatec Plus TSC. Such Al could still be detected after 5-min ultrasonic cleaning[12]. Lorente *et al.* also detected Al on zirconia surfaces treated with 30-µm Cojet (3M) TSC[15]. They found that the amount of alumina present on the zirconia surface decreased after ultrasonic cleaning for 10 min. This study clearly revealed that Al particles remained despite a strong air blown. Some Al particles were fractured, by which fragments remained attached onto the zirconia surface.

Amaral *et al.* claimed that both laboratory and chairside TCS led to a high and durable bond strength to zirconia[24]. Other studies however demonstrated that surface-conditioning methods, such as alumina sandblasting and Rocatec TSC, did not significantly affect the bonding effectiveness of the composite cements Panavia F (Kuraray Noritake Dental) and RelyX Unicem (3M) to zirconia[7]. Bonding effectiveness is known to depend on many factors, such as micro-mechanical interlocking, chemical adhesion, surface characteristics, and the actually applied adhesives[25]. TSC with Rocatec/Cojet sand has a double effect, namely micro-roughening and silica coating. Therefore, even when the zirconia surface is not completely covered by a silica coating layer, micro-mechanical interlocking will still contribute to the bond strength to zirconia[26,27].

The combined 10-MDP/silane-containing Clearfil Ceramic Primer was found to result in a significantly higher bond strength to TSC-treated zirconia than that achieved when TCS-treated zirconia received silane coupling *via* ESPE Sil. In our study, the zirconia area exhibiting a silica coating layer was incomplete, so that also the silane coupling effect may have been incomplete. On the other hand, the functional monomer 10-MDP has been shown to chemically interact with zirconia[9]; 10-MDP does not chemically bond to a silica-coated zirconia surface. Therefore, 10-MDP must have chemically interacted with the zirconia surface zones that were not coated with a silica layer; these zones were probably prevailing, explaining the better shear bond strength recorded with Clearfil Ceramic Primer than with ESPE Sil. Some authors indeed recommended the combination of Rocatec/Cojet TSC with the application of a 10-MDP-containing primer, so to enhance the zirconia-bonding

properties[9,28]. Although our data confirmed better results using this approach, this positive effect is inconsistent with the working principle of TSC; it is supposed to coat the zirconia surface with silica that in turn would not allow 10-MDP to bond.

CONCLUSION

Ultrastructural characterization of TSC-treated zirconia revealed that TSC incompletely coated zirconia with silica and alumina. A 10-MDP/silane-containing primer was found to bond strongly with TCS-treated zirconia thanks to the chemical interaction of 10-MDP with the zirconia surface zones that remained uncoated with silica.

ACKNOWLEDGMENTS

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