

Evaluation of an Innovated Zirconia Surface Treatment Technique

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Abstract: *Objectives:* To introduce a new surface treatment of pre-sintered zirconia silica coating to modify zirconia surface and to evaluate its effect on certain properties. *Materials and Methods:* A total of 104 pre-sintered zirconia specimens were prepared and divided into three groups: (1) Control (C) (2) Treated before sintering by an innovated silica containing gel (PCT/EG2011/000014) (T) (3) Airborne particle abrasion after sintering (A). Specimens' surfaces were examined using Scanning Electron Microscopy (SEM) and chemical analyzed with Energy Dispersive X-ray Analysis (EDXA). Crystalline structures were characterized by X-ray Diffraction (XRD). The specimens were tested mechanically for biaxial flexural strength and indentation fracture toughness. *Results:* Pre-sintered zirconia showed a porous structure which was obviously reduced after sintering in group C. Presence of glassy layer was evident in group T. Group A showed surface scratches. Both groups T and A showed higher surface roughness values compared to group C. Elemental analyses of group C and A were found to be mainly zirconia. Other elements were evident in group T. Silica powder used in group T showed an amorphous structure before sintering transferred to cristobalite after sintering, in addition to tetragonal zirconia. After abrasion, monoclinic peaks appeared in group A. Group T showed highest flexural strength, while group A showed highest fracture toughness. *Conclusions:* Pre-sintered zirconia silica coating is an easy effective technique for modifying zirconia surface without negatively affecting the strength.

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1. Introduction:

Developments over the last ten years in ceramic materials science have led to a class of high strength materials such as alumina and zirconia based ceramics⁽¹⁾. Utilizing zirconia in the dental field opens the design and application limits of all-ceramic restorations⁽²⁾.

The unique mechanical properties of zirconia allow its use as a core material for long span all-ceramic restorations. On the other hand, its high surface stability and chemical inertness represents a difficulty in establishing a strong and reliable bond with the underlying cements. This adversely affects its retention and stability.⁽³⁾

Generally, bonding of many all-ceramic restorations to resin cements is usually increased via micro-mechanical attachment e.g. hydrofluoric acid etching and chemical bonding e.g. silane coupling agent. Nevertheless, the zirconia being non-glass containing ceramic, these techniques are ineffective.⁽⁴⁾

Several surface roughening and coating methods have been used to optimize the surface of zirconia, in an attempt to improve its bonding with resin cements. These included tribochemistry (sandblasting with

silica-coated aluminum oxide particles)⁽⁵⁾, silicoating (pyrolytically applying a silica coating or using PyrosilPen-Technology⁽⁶⁾, plasma spraying of hexamethyldisiloxane⁽⁷⁾ and selective infiltration-etching technique (zirconia was heated to 750°C for 2 minutes, cooled to 650°C for 1 minute, reheated to 750°C for an additional 1 minute, and then cooled to room temperature)⁽³⁾.

Unfortunately, these trails insignificantly increase zirconia-resin bond strength, decrease zirconia mechanical properties or require several technical complicated steps which also consume long time.

Although surface treatment of pre-sintered zirconia might be less complicated, yet it is not commonly investigated. Therefore, a trial to modify the surface of pre-sintered zirconia rather than sintered zirconia was conducted in this research. Evaluation the effect of this treatment on certain physical properties of sintered zirconia as regard surface characteristics, flexure strength and fracture was performed.

2. Materials and Methods

2.1. Specimen Preparation and Surface Treatment

A total of 104 disc-shaped pre-sintered zirconia specimens (19mm diameter x 1.5mm thickness) were prepared from yttrium partially stabilized zirconia blocks (In-Ceram YZ 20/19, VITA Zahnfabrik, Germany). The specimens were randomly divided into three main groups: (1) *Control* (C) (2) Treated before sintering by coating with an innovated silica containing gel (PCT/EG2011/000014) (T) (3) *Airborne particle abrasion* after sintering (A). After sintering, the specimens (15mm diameter x 1.2mm thickness) were subjected to veneering temperature firing cycles without actual veneer building to simulate the actual laboratory procedure.

2.2. Chemical Analysis of Zirconia Blocks and Silica Powder

Chemical compositions of the zirconia blocks and the silica powder were analyzed by X-Ray Fluorescence Spectrometer (XRF), (Axios, Panalytical, Netherland).

2.3. Surface Microstructure

Scanning Electron Microscope (SEM), (Supra 40, Carl Zeiss NTS GmbH, Germany), was used to examine the specimens' surface microstructures. Pre-sintered and sintered zirconia specimens from the control, treated and abraded groups (C, T, A respectively) were mounted on coded brass stubs. The specimens were sputter coated with 10Å gold platinum and observed at 5000× magnification for both top and lateral views (for each surface; two for top views and two for lateral views). An accelerating voltage of 20.0 kV to 30.0 kV was used.

2.4. Surface Metrology:

2.4.1. Surface Roughness:

Surface profiles of C, T, A specimens were determined using confocal laser scanning microscope (True Cofocal Scanner, Leica TCS SP2, Germany). A 458 nm Argon laser (1mW) was used as a light source, and the specimens were observed at 20× magnification. The measuring area was 1.5 μ×1.5 μ and the height of the z-stack was 30 μm in 1μm intervals. Measurements were performed at three different areas for each specimen (n=5/group).

2.4.2. Coat Thickness in T Group:

Five specially prepared specimens were used for measuring the coat thickness in the treated group (T). The silica coating material was applied in a strip form in the middle portion of each specimen, *figure 1*.

The coat thickness in group T was measured by both the non-contact confocal laser scanning microscope and a contact stylus surface profiler (Taylor Hobson Precision, USA). Using the confocal laser microscope, a line was drawn across the coated part, starting and ending by the control untreated surfaces at both sides. In case of the contact stylus method, *the stylus* of the surface profiler traced the surface passing through the coated part.

Measurements were performed at three different locations for each specimen. Graphs were obtained; the mean coat thickness was calculated.

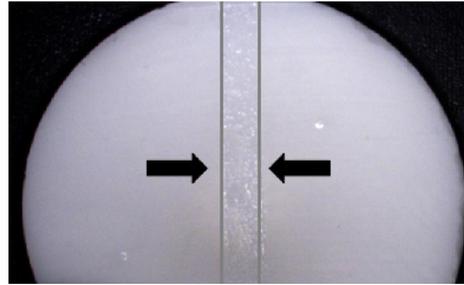


Figure (1): Specimen for coat thickness measurement.

2.5. Surface Elemental Analysis:

Elemental analysis of the specimens' surfaces was obtained using Energy-Dispersive X-ray Spectroscopy (EDX), (Supra 40, Carl Zeiss NTS GmbH, Germany). The constituents of the materials at the surface were detected with an accelerating voltage of 20- 30kV and a magnification of 20000×.

2.6. Crystalline Phase Identification:

X-ray diffraction (XRD) analyses were conducted to identify the crystalline phases of the pre-sintered zirconia and the silica powder used for coating. The crystallographic structures of the sintered specimens from groups C, T, A was analyzed before and after the veneering temperature firing cycles, using *X-Ray Diffractometer*, (*X'Pert*, *Philips*, *Netherland*). The surfaces of the specimens were scanned with copper X-unit (Cu Kα) X-ray from 0 to 80° 2θ degrees with a step size of 0.05 and 2 seconds step interval. The relative amount (X_M) of the monoclinic phase within the zirconia phases was calculated as suggested by **Garvie and Nicholson** ⁽⁸⁾

2.7. Flexure Strength Test:

To determine the effect of the different surface treatments on strength properties of the ceramic materials, the biaxial flexural strength test (piston on three balls) was used, as described in the ISO standard 6872 for dental ceramics, (n=10/group).

A universal testing machine (Sintec 2/G, MTS system, USA) was used at a speed of 1mm/min with a crosshead speed of 0.15mm/min. To support the test specimen, three steel balls with a diameter of 3.2mm were positioned 120 degrees apart on a support circle with a diameter of 10mm. The disc shaped specimens were positioned concentrically on these supports and the load was applied at the centre of the specimen with a flat punch 1.4 mm in diameter.

The load at the point of fracture was recorded, and the biaxial flexural strength for each specimen was calculated with the following equation: ⁽⁹⁾

$$S = -0.2387 P (X-Y)/d^2$$

Where S = the maximum centre tensile stress (MPa) (the flexural strength at fracture), P = the total load causing fracture (N),

$$X = (1+\nu) \ln (r_2/r_3)^2 + [(1-\nu)/2] (r_2/r_3)^2, Y = (1+\nu) [1 + \ln (r_1/r_3)^2] + (1-\nu) (r_1/r_3)^2$$

In which, ν = Poisson's ratio, r_1 = the radius of the support circle, r_2 = the radius of the loaded area (mm), r_3 = the radius of the specimen (mm), d = the specimen thickness at the origin of fracture (mm).

For this study, $\nu = 0.25$, $r_1 = 5$ mm, $r_2 = 0.7$ mm and $r_3 = 7.5$ mm.

2.8. Indentation Fracture Toughness Test:

Fracture toughness for the different groups (C, T, A) was determined by the indentation technique proposed by Anstis *et al.* ⁽¹⁰⁾ ($n=10/\text{gp}$). Indentations were performed with a standard Vickers diamond pyramid on a hardness testing machine (Vickers; Instron Wolpert, UK).

According to Anstis *et al.* ⁽¹⁰⁾, the crack length, c (as measured from the center of the indent), should be at least equal to or greater than the diagonal length ($2a$). The standard Vickers loads of the hardness instrument were evaluated first to determine the optimum load to meet the criterion of $c/a \geq 2$. Therefore, it was determined by trial and error that a load of 490 N should be used to produce a c/a ratio of 2.

Readings were recorded for each group under the Scanning Electron Microscope (SEM) and the fracture toughness was calculated as follows ⁽¹⁰⁾:

$$K = 0.016 \times (E/H)^{1/2} \times P/c^{3/2}$$

Where, K = the fracture toughness of the material ($\text{MPa} \times \text{m}^{1/2}$), E = the elastic modulus, $H=P/2a^2$, P = the load applied (N), a = the indent half diagonal (m), c = the crack length measured from the center of the indent (m).

3. Results

3.1. Chemical Analysis of Zirconia Blocks and Silica Powder

The chemical analysis of the zirconia blocks and the silica powder revealed the presence of many minor elements not mentioned by the manufactures. The zirconia blocks consisted of the following oxides in wt%: ZrO_2 (91.69), Y_2O_3 (5.08), HfO_2 (0.23), Nd_2O_3 (2.26), IrO_2 (0.15), Bi_2O_3 (0.05), SiO_2 (0.09), Al_2O_3 (0.03), MgO (0.03) and SO_3 (0.002). Due to heating, there was a loss on ignition (L.O.I) about 0.38 wt %. While, the composition of the silica powder in wt % was as follows: SiO_2 (94.49), Na_2O (1.14), CaO (0.14), MgO (0.16), Al_2O_3 (0.11), Fe_2O_3 (0.06), K_2O (0.01), NiO (0.01), P_2O_5 (0.01), SO_3 (0.004) and L.O.I. (3.87).

3.2. Surface Microstructure

The pre-sintered zirconia specimens revealed a highly porous microstructure (*Figure 2*). While after sintering, group C exhibited a densely grained structure with an obvious reduction in porosity and increase in grain size (*Figure 3*). Presence of a glassy layer over the zirconia grains was evident in group T with limited penetration (*Figure 4*). The airborne particle abrasion (group A) resulted in creation of a highly rough surface with surface scratches (*Figure 5*).

3.3. Surface Metrology:

3.3.1. Surface Roughness:

Both groups T and A showed significantly higher mean surface roughness values ($14.1 \mu\text{m}$ and $14.9 \mu\text{m}$ respectively) compared to group C ($6.8 \mu\text{m}$).

3.3.2. Coat Thickness in T Group:

The means of coat thickness in group T measured by confocal microscope and stylus surface profiler were $8.97 \mu\text{m}$ and $9.16 \mu\text{m}$ respectively. There was no significant difference between the confocal and the stylus measurements.

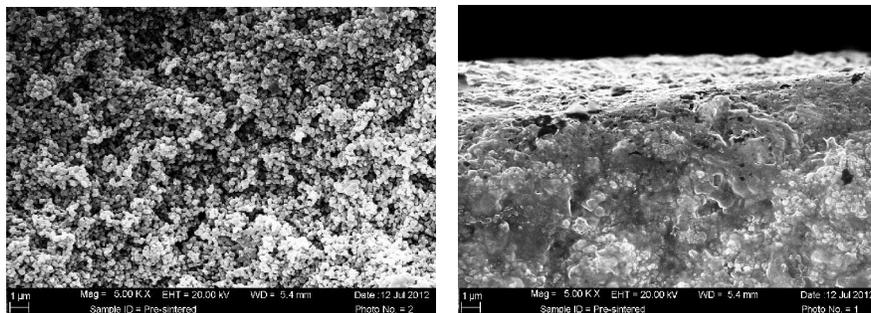


Figure (2): SEM micrograph of pre-sintered zirconia specimen.
(a) Top view, (b) Lateral view, Magnification: 5000 \times .

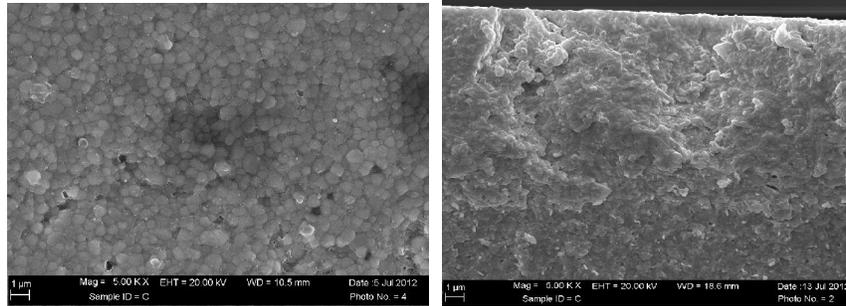


Figure (3): SEM micrograph of control specimen (C).
(a) Top view, (b) Lateral view, Magnification: 5000 \times .

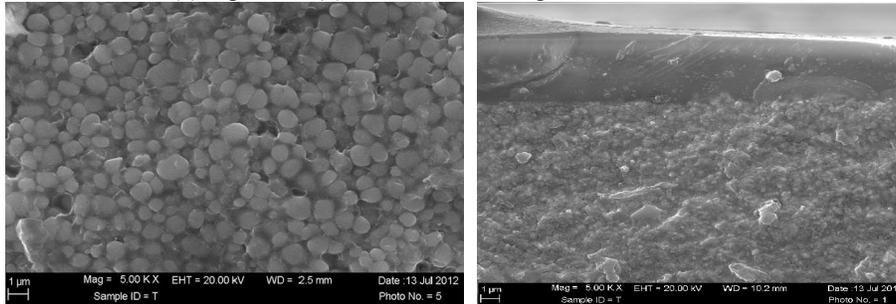


Figure (4): SEM micrograph of treated specimen (T).
(a) Top view, (b) Lateral view, Magnification: 5000 \times .

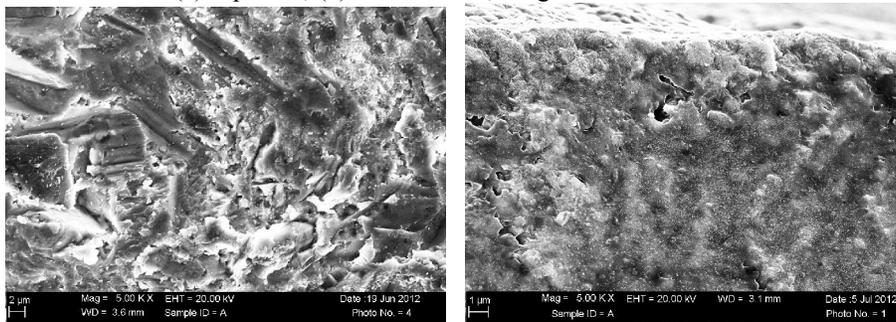


Figure (5): SEM micrograph of treated specimen (T).
(a) Top view, (b) Lateral view, Magnification: 5000 \times .

3.4. Surface Elemental Analysis:

Elemental analyses of the pre-sintered zirconia and specimens from groups C and A were found to be mainly zirconia. On the other hand, in specimens from group T, other elements in addition to zirconia were evident as silica, sodium, magnesium, aluminum and calcium.

3.5. Crystalline Phase Identification:

The pre-sintered zirconia specimens revealed both tetragonal phase with some monoclinic phase of 4.5%. While after sintering, only tetragonal zirconia was identified in group C. On the other hand, the silica powder used in coating group T showed an amorphous silica structure before sintering, which transferred to cristobalite after sintering. In addition, tetragonal zirconia phase was also detected. After air particle abrasion, monoclinic peaks appeared (8.6%)

in group A. The veneer firing cycles did not induce phase transformation.

3.6. Flexure Strength Test:

Group T showed the highest significant mean value (982.4 MPa), followed by group C (846.2 MPa), and then group A (482.1 MPa).

3.7. Indentation Fracture Toughness Test:

Group A showed the highest significant mean fracture toughness (6.6 MPa \times m^{1/2}), followed by group T (5.2 MPa \times m^{1/2}), and then group C (3.9 MPa \times m^{1/2}).

4. Discussion

Scanning electron microscope of the pre-sintered zirconia showed a porous structure, *figure 2*, which was obviously reduced in the control group

(C), *figure 3*, due to the sintering process. The SEM of the treated group (T), *figure 4*, showed a glassy coat upon the zirconia surface preserving the zirconia grain structure as that in group C. In the abraded group (A), *figure 5*, surface flaws were detected which may affect the mechanical properties. The surface damage after the airborne particle abrasion process was in agreement with **Aboushelib MN *et al***⁽³⁾ and **Moon JE *et al***⁽¹¹⁾.

It was shown that both the treated group (T) and the abraded group (A) showed significantly higher surface roughness values compared to the control group (C). This significant increase in the surface roughness in group T may be attributed to the rough glassy coat after treatment. While in group A, this may be attributed to the formed surface flaws as a result of airborne particle abrasion. The significant variation in the surface roughness between the airborne particle abraded and control specimens was in agreement with **Castillo de Oyagüe R *et al***⁽¹²⁾. However, this finding was contradictory to **Moon JE *et al***⁽¹¹⁾ who found insignificant difference in the surface roughness between the abraded and control specimens.

Test protocol variation may be the cause for this contradiction in the literature regarding the surface roughness of the airborne-particle abraded specimens. Variables may include zirconia type⁽¹³⁾, bombardment particle size⁽¹⁴⁾, standoff distance of airborne-particle abrasion, shooting pressure, time taken and cleansing procedure⁽¹¹⁾.

The coat thickness in the group T was measured by both the confocal laser scanning microscope and the stylus surface profiler to compare the non-contact and contact techniques respectively. Specially prepared specimens were used; the coat in the middle portion and control surfaces at both sides. The aim was to accurately adjust the graph level after coat thickness measurement by having zero starting and ending points. This design prevented any misleading data which may occur due to specimen tilting or minor discrepancies in the specimen surface during preparation.

The coat thickness should allow complete seating and fitness of the dental restorations. In the literature, it was reported that the marginal and internal gaps of the zirconia dental restorations ranged from 29 to 119µm, depending on the milling system used to manufacture the Y-TZP frameworks⁽¹⁵⁾. The mean values for coat thickness measured by the confocal microscope and the stylus were 8.97µm and 9.16µm respectively. There was insignificant difference between the values obtained by both methods. The coat thickness in the group T was supposed to be lower than the reported internal and

marginal gaps, which would not interfere with the seating and fitness of the restoration.

The elemental analyses of the pre-sintered zirconia and specimens from groups C and A by the EDX were found to be mainly zirconia. However, the other elements present in the composition were not detected. This might be due to EDX limitation, which could not detect accurately elements with concentration equal or less than 5 wt%⁽¹⁶⁾. The treated group (T) showed the presence of silica, sodium, magnesium, aluminium, calcium in addition to zirconia. Yet, their percentages were not clearly identified from the curve which may be due to EDX limitation⁽¹⁶⁾.

The crystalline structure of the pre-sintered zirconia specimens by XRD showed a relative monoclinic phase (4.5%) in addition to the tetragonal one. This was in agreement with **Tsalouchou E *et al***⁽¹⁷⁾ who found that the zirconia powder before sintering revealed tetragonal and monoclinic phases. However, this finding was contradictory to **Moon JE *et al***⁽¹¹⁾ who stated that the pre-sintered Y-TZP ceramics consisted of almost 100% tetragonal structures.

After sintering, the tetragonal phase only was identified in the control group (C). This result was assisted by **Tsalouchou E *et al***⁽¹⁷⁾ and **Moon JE *et al***⁽¹¹⁾ who noticed that there were no monoclinic structures in the as-sintered Y-TZP. This might be explained by the monoclinic-tetragonal phase transformation induced by the sintering temperature, with an end temperature 1530°C. It was known that heating the zirconia above 1170°C, phase transformation from monoclinic to tetragonal occurred⁽¹⁸⁾.

The relative monoclinic phase after the airborne-particle abrasion was 8.6% in group A. This may be attributed to the stress generated on the zirconia surface by the abrasion process which accelerated the tetragonal- monoclinic transformation^(19, 20). This finding was assisted by the results of **Moon JE *et al***⁽¹¹⁾ who revealed the appearance of monoclinic phase after the sandblasting procedure (11.4%).

The silica powder used in group T revealed an amorphous structure. However after sintering, crystallization into cristobalite was detected. This might be caused by the sintering temperature and time (seven and a half hours with an end temperature 1530°C) which may provide the energy required for crystallization. It was known that cristobalite silica was the detected crystal structure at temperature above 1470°C until the fusion of silica at 1710°C⁽²¹⁾.

The crystallographic structures of the different specimens were analyzed both before and after the veneering cycles to detect any crystalline changes in either the zirconia or silica. However, no phase

transformation was detected after these cycles. This may be explained on the basis that the veneering firing temperature (910°C maximum) might not induce phase transformation. It was known that above 1170°C, zirconia transformed into the tetragonal, and at 2370°C, the material changed into a cubic phase⁽¹⁸⁾.

The treated group (T) showed significantly the highest flexural strength (982.4MPa). This may be attributed to the addition stress required to fracture the coat and the sealing of any existed flaws by the glassy layer. On the other hand, the abraded group (A) showed significantly the lowest mean flexural strength (482.1 MPa). The detected surface flaws and monoclinic phase transformation might be the cause of decreasing the flexural strength in group A. The reduction in flexure strength after air borne particle abrasion was in agreement with previous results which related the reduction to the degree of surface damage⁽²²⁻²⁴⁾. On the other hand, some studies reported an increase in strength after airborne particle abrasion and related such finding to the creation of compressive fields as a result of the induced tetragonal–monoclinic transformation of the surface crystals⁽²⁵⁻²⁸⁾. Moreover, Wang H *et al*⁽²⁹⁾ indicated that particle abrasion with 50µm aluminium oxide resulted in an increase in the strength of zirconia, possibly by removing weakly attached surface grains and by the elimination of milling and grinding trace lines. This controversy might be attributed to the test protocol variation mentioned previously.

The abraded group showed significantly the highest mean fracture toughness (6.6 MPa x m^{1/2}). This might be attributed to the compressive stress layer which had been formed due to the monoclinic phase transformation on the zirconia surface that counteracted the residual tensile stresses from the cracks induced by the Vicker indenter. The treated group showed higher mean fracture toughness (5.2 MPa x m^{1/2}) than the control group (3.9 MPa x m^{1/2}). This may be related to the mechanical response of the coated system, where the crack energy was dissipated by both the coat and the zirconia substructure.

However, the data obtained from the Vicker indentation fracture toughness was contradictory to what obtained by the flexure strength test. The former test measured only the surface properties and hence may be misleading. It was stated in a previous study⁽¹⁴⁶⁾ that the Vicker indentation fracture toughness technique was not reliable as a fracture toughness test for ceramics or for other brittle materials as fracture resistance cannot be readily defined. Thus, it was recommended that the Vicker indentation fracture toughness technique no longer to be acceptable for the fracture toughness testing of ceramic materials⁽¹⁴⁶⁾.

Conclusions:

Within the limits of this investigation, the following conclusions could be drawn:

1. Surface treatment of pre-sintered zirconia represents a simple, time saving and non-complicated method to modify the sintered zirconia surface, compared to the conventional techniques.
2. Silica coating of pre-sintered zirconia is a valuable technique to enhance the zirconia-resin bond using silanation and conventional resin cement.
3. The flexure strength test rather than the Vicker indentation fracture toughness test is considered to be a more reliable method in evaluating the mechanical behavior of the different surface treated zirconia.
4. Coating of pre-sintered zirconia with silica increases the flexure strength of the sintered zirconia.
5. Air borne particle abrasion after sintering has a negative influence on the strength and induces phase transformation of the zirconia.

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