Effect of sandblasting on surface roughness of zirconia-based ceramics and shear bond strength of veneering porcelain

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This study aims to investigate the effect of sandblasting on the surface roughness of zirconia and the shear bond strength of the veneering porcelain. Pre-sintered zirconia plates were prepared and divided into four groups. Group A were not treated at all; group B were first sandblasted under 0.2 MPa pressure and then densely sintered; group C and D were sintered first, and then sandblasted under 0.2 MPa and 0.4 MPa pressures respectively. Surface roughness was measured and 3D roughness was reconstructed for the specimens, which were also analyzed with X-ray diffractometry. Finally after veneering porcelain sintering, shear bond tests were conducted. Sandblasting zirconia before sintering significantly increased surface roughness and the shear bond strength between zirconia and veneering porcelain (p<0.05). Sandblasting zirconia before sintering is a useful method to increase surface roughness and could successfully improve the bonding strength of veneering porcelain.

Keywords: Zirconia, Sandblasting, Shear strength, Veneering porcelain

INTRODUCTION

Ceramics are widely used in prosthetic and operative dentistry1-5) with the advent of new technologies such as heat press, injection mold, slip cast, computer-aided design or computer-aided manufacturing and glass infiltration. Yttria stabilized tetragonal zirconia polycrystal (Y-TZP) bioceramic inert is such ceramics with good biocompatibility, mechanical properties and aesthetics6-8), and has been successfully used as a core material in full-ceramic dental restorations. In the final prosthetic process, veneering porcelain is deposited on zirconia-based ceramics, but long-term clinical results for zirconia all-ceramic restorations need to further study9-11), which are attributed to debonding or fracture of veneering porcelain. So various surface modification techniques have been proposed to improve the bonding strength between zirconia and veneering porcelain12-17), such as sandblasting with different particle sizes and different oxides, mechanical grinding, application of hydrogen fluoride, and heat-treatment. However, some of these techniques may certainly damage the outer surfaces of zirconia-based ceramics and extremely thin frames12,13,16,17). For example, sandblasting surface treatment may trigger the phase transformation from tetragonal to monoclinic structures, causing strength reduction and fracture in zirconia because of the loss of transformation capacity during critical loading, and reducing the lifespan of zirconia ceramics16,19). This study aims to investigate how different manners of applying sandblasting affect the surface roughness of zirconia and the shear bond strength of the veneering porcelain.

MATERIALS AND METHODS

Specimen preparation
Prefabricated zirconia blocks (Highly transparent, Nissin-Metec, Shanghai, China) which consisted of ZrO_2+HfO_2+Y_2O_3+Al_2O_3>99.9%, and other oxides<0.1% were cut with a diamond saw (Isomet 4000 Linear Precision, Buehler, Chicago, USA) into 16 mm×16 mm×3 mm plates under copious water (n=11), and then the surface of each disc was polished with water-proof silicone-carbide paper until 1,000#. Next, the specimens were randomly divided into 4 groups (see Table 1), and group B–D were sandblasted using 100 mesh alumina particles under different blasting pressures (0.2 MPa for group B and C and 0.4 MPa for group D) for 10 s from a distance of 10 mm by a sandblasting instrument (TJK-BP II, Tianjin Haide, Tianjin, China), and sintered in a programmable furnace (ZSK 1700, Cinite, Beijing, China) at 1,450°C sintering temperature for 2 h.

Surface roughness
Surface roughness was measured using a surface-texture and contour measuring instrument (JB-4C, Shanghai Taiming Optical Instrument, Shanghai, China) whose stylus has 2 µm tip radius and 4 mm traversing length. The cut-off value of the instrument was set to be 0.8 mm. For each specimen, surface roughness was measured at 4 different locations, and these measured values were averaged to obtain the average surface roughness (Ra).

The 3D roughness of one specimen in each group was also reconstructed using scanning electron microscopy (Phenom proX, Phenom-World BV, Eindhoven, Holland) to verify that the scratches have similar depths and widths.

Color figures can be viewed in the online issue, which is available at J-STAGE.
Received Jan 3, 2014: Accepted Jul 7, 2014
doi:10.4012/dmj.2014-002   JOI JST.JSTAGE/dmj/2014-002

Dental Materials Journal  2014; 33(6): 778–785
Table 1  Surface treatments for Y-TZP

| Group | N | Parameter                                      |
|-------|---|------------------------------------------------|
| group A | 11 | without sandblasting                           |
| group B | 11 | sandblasted under 0.2 MPa pressure and then sintered |
| group C | 11 | sintered and then sandblasted under 0.2 MPa pressure |
| group D | 11 | sintered and then sandblasted under 0.4 MPa pressure |

**Phase analysis**

Before porcelain fusing, the zirconia specimens were examined by X-ray diffractometer (XRD 7000 X-ray diffractometer, Shimadzu, Kyoto, Japan) with Cu-ka radiation (λ=1.5406 Å). The experimental conditions were as follows: 2θ range of 25–35° was scanned at 2°/step and with 6 s photon counting time per step. The relative mass fraction of monoclinic phase (X_m) on specimen surfaces was calculated using Garvie and Nicholson’s formula:

\[
X_m = \frac{I_m(1\bar{1}1)+I_m(111)}{I_m(1\bar{1}1)+I_m(111)+I_t(101)}
\]

where \(X_m\) is the relative mass fraction of monoclinic phase, \(I_m(1\bar{1}1)\) is the intensity of monoclinic peak at 28.2°, \(I_m(111)\) is the intensity of monoclinic peak at 31.5°, and \(I_t(101)\) is the intensity of tetragonal peak at 30.2°.

**Shear bond tests**

Specimens of each group were veneered with porcelain Cerabien ZR (Noritake, Nagoya, Japan) according to manufacturers’ instructions. A porcelain layer is first sintered on each ceramic specimen. A metal tube (6 mm height, 12.0 mm outer diameter, and 4.5 mm inner diameter) was placed on the center of each specimen, and the shade base powder of zirconia was kneaded with attached liner liquid inside the tube to build up a 1 mm thick layer, which was then fired together with the specimen in an automatic furnace (LECTRA, Ugin Dentaire, France). After firing, the height of this layer of shade-base buildup was adjusted to be 0.5 mm by grinding with rotary tools, and the thickness of ceramic layer was checked by a caliper. Next, a second layer up to a thickness of 4 mm was constructed in the same procedure described above and shown in Fig. 1. The heating conditions for both are also shown in Table 2.

To evaluate shear bond strength, each specimen was embedded in an acrylic mold using methyl methacrylate resin, and shear bond tests were conducted on a universal testing machine (AG-X Plus, Shimadzu, Kyoto, Japan). The machine applied an increasing shear load to the specimen at 1 mm/min crosshead speed until the veneering porcelain was separated from the zirconia-based ceramic. The shear bond strength was then derived by taking the average of 10 measured data.

**Zirconia-veneering porcelain interfacial analysis**

After porcelain sintering, one zirconia-veneering porcelain sample from each group was invested in an acrylic resin. After 24 h storage at room temperature, they were cut with low speed electrosection and the zirconia-veneering porcelain interfaces were exposed. Next, these interfaces were ground with SiC paper under continuous water cooling, and to the grit size was improved from 600 to 2,000. They were also polished with 0.25 µm diamond paste on a grinding/polishing machine. Finally, the polished samples were cleaned in distilled water by ultrasonic cleaner for 10 min and sputter-coated with gold in a sputter-coating unit (SCD 004 Sputter-Coater with OCD 30 attachment, Bal-Tec, Vaduz, Liechtenstein). After sputter-coating with gold, the interfaces were examined under a scanning electron microscope (S-3400N, Hitachi, Tokyo, Japan) with 400×magnification.

**Failure mode analysis**

The failure between the zirconia and veneering porcelain was defined as ‘adhesive’. The failure within either the zirconia or veneering porcelain was defined as ‘cohesive’. The term ‘mixed’ failure was used to describe the combination of these two failure types. The fractured surfaces were observed by scanning electron microscope with 245×magnification.

**Statistical analyses**

The results are expressed in the form of “mean±SD”
for each parameter, and one-way analysis of variance and Tukey’s HSD pairwise multiple comparisons were carried out to compare different surface treatments. A value of $p<0.05$ was considered statistically significant.

**RESULTS**

**Surface roughness**

Comparison of surface roughness is shown in Fig. 2. The average surface roughness (Ra) for group A, B, C, and D are $0.63\pm0.42$ µm, $4.65\pm1.01$ µm, $0.89\pm0.35$ µm and $1.53\pm0.38$ µm, respectively. The Ra of group B is much greater than and significantly different from that of group A, C, and D ($p<0.001$), indicating that sandblasting under 0.2 MPa pressure before sintering produces more uneven surface. Group D also has a Ra significantly different from that of group A ($p<0.05$).

The reconstructed images of 3D roughness representative of each group are shown in Fig. 3, and group B shows more irregular profiles (Fig. 3b). The softer zirconia surface before sintering in group B seems to be affected more by sandblasting than the sintered zirconia in group C and D.

**Surface characterization analysis of specimens**

The XRD (X-ray diffractometry) patterns shown in Fig. 4 indicate that group A and B only have tetragonal structures, but group C and D have both tetragonal and monoclinic structures. The relative amount of monoclinic phase was 1.64% for group C and 2.20% for group D, with marked features of $(1\bar{1}1)$ and $(111)$ in the XRD pattern.

**Shear bond tests**

Figure 5 shows the result of shear bond strength test. Group B has the highest strength of 35.02±3.18 MPa, significantly higher than those of group A and C ($p<0.001$ and $p=0.001$, respectively). The strengths of others decrease in the order group D (29.82±5.78 MPa)>C (25.04±4.78 MPa)>A (20.97±6.27 MPa), with the strength of group A significant different from that of D ($p=0.002$).

**Zirconia-veneering porcelain interfacial analysis**

Figure 6 shows the SEM micrographs of the zirconia-veneering porcelain interface of each group. The compacted interface appears to be straight between zirconia and the veneering porcelain in the SEM micrographs of groups A and C, but irregularly wavy for group B and D. The interface of group B looks even bumpier than that of group D.

**Failure mode analysis**

Failure types and distribution are presented in Table 3. After shear bond testing, every group showed mixed type of failures, especially in group C and D, they failed entirely mixed type. However, adhesive fractures at zirconia-veneering porcelain interface and cohesive fractures in veneering porcelain were observed in group A and B respectively. None of the specimens fractured
Fig. 3  The representative images of 3D of each group specimens.
(a) specimen of group A; (b) specimen of group B; (c) specimen of group C; (d) specimen of group D.

Fig. 4  XRD patterns obtained for each group.
A-specimen of group A; B-specimen of group B; C-specimen of group C; D-specimen of group D.
"t" represents the tetragonal phase and "m" represents the monoclinic phase.

Fig. 5  Measured shear bond strength of each group.
A-specimen of group A; B-specimen of group B; C-specimen of group C; D-specimen of group D.
Vertical bars indicate the standard deviation and identical letters indicate that the values are not significantly different ($p>0.05$).
within the zirconia. Figure 7 shows the SEM micrographs of the zirconia-veneering porcelain fractured surfaces of each group. There is adhesive fracture at zirconia-veneering porcelain interface of group A, almost no porcelain components remained in zirconia surface. While cohesive fracture within the veneering porcelain for group B, more porcelain components remained in zirconia surface. For group C and D, both zirconia and porcelain components were observed.

**DISCUSSION**

Veneering porcelain fused to dental metal, generally employing both mechanical and adhesive retentions, has been well studied, and the bonding strengths are predictable. However, zirconia is acid-resistant or non-etchable, and non-conducting, and has high mechanical properties, thus surface roughening treatments have been recommended to improve the bonding strength between zirconia and veneering porcelain by creating micromechanical interlocking. Current available roughening techniques are: (1)
grinding, (2) abrasion with diamond (or other) rotary instruments, (3) air abrasion with alumina (or other) particles, (4) acid etching (typically HF), and (5) combination of any of these techniques. Unfortunately, the composition and physical properties of zirconia make it difficult to roughen the zirconia surface for mechanical retention. Moreover, zirconia differs from conventional silica-based materials like porcelain, and does not readily etched by HF. Thus, more aggressive mechanical abrasion methods are required to increase surface roughness, which, however, possibly create surface flaws and reduce the strength of material.

Sandblasting is a useful method, however, it is considered that sandblasting may put stress on zirconia surfaces and accelerate tetragonal-to-monoclinic (t→m) phase transformation, and air particle abrasion should not be used, particularly with zirconia, because it might cause microfractures that would reduce functional strength and lead to premature and catastrophic failure. In-vitro analysis has shown that surface flaw generation can reduce the fracture strength of zirconia significantly.

In this study, sandblasting before sintering (group B) is found to improve surface roughness by over 500% compared with the same condition of sandblasting after sintering (group C), indicating that sandblasting zirconia before sintering could be a very useful method to improve surface roughness. This could be explained by the much lower hardness of zirconia before being sintered, which resulted in rougher zirconia surface through sandblasting, thus larger surface area available for mechanical interlocking.

The XRD patterns of group C and D revealed monoclinic phase. In group B, sandblasting might produce monoclinic phase, but the phase was not detected after sintering, reverse transformation monoclinic-to-tetragonal (m→t) could have occurred during the later sintering process.

To estimate bond strength, there are many methods such as shear bond tests, bending tests, and tensile tests with push-out tests are used. In this study, shear bond tests were chosen due to not only many reports in the same experimental category but severe testing method. The specimens sandblasted before sintering has larger surface roughness as shown in Fig. 3, thus larger surface area, the larger surface area leads to greater shear bond strength than those of the control group. This relation is also supported by the observed zirconia-veneering porcelain interfaces shown in Fig. 6. The interface of group B appears distinctly most wavy, thus having largest surface area connected with the veneering porcelain. However, surface roughness of group B is three times bigger than that of group D, there is no significant difference between them about shear bond strength. This phenomenon may be explained as follows, firstly, the relation between surface roughness and shear bond strength may not be linear relationship; secondly, excessive rough surface may lead to stress concentration which may consequently weaken the interfacial bonding between zirconia and veneering porcelain. However, sandblasting may result in a possible contamination of the zirconia substrates by...
the alumina particles, which may consequently weaken the interfacial bonding between zirconia and veneering porcelain. Due to the small plastic deformation of zirconia, considerably less alumina particles remained on zirconia surface during sandblasting. Moreover, with ultrasonic cleaning and compressed air drying for zirconia surface after sandblasting, there were more less remnants of the alumina particles. Therefore we did not put the residual alumina as the main factors affecting the bonding strength between zirconia and veneering porcelain.

The failure mode results suggested that all groups mainly exhibited mixed failure types. Cohesive failure within veneering porcelain occurred only in group B, this could be explained by the improved surface contact between the zirconia and veneering porcelain. While in group A, the failure type obtained adhesive type due to the relatively smooth surface.

Within this limited study, the experimental results show that sandblasting zirconia before sintering is a useful method to increase surface roughness and could successfully improve the bonding strength between zirconia and veneering porcelain.

CONCLUSION

Sandblasting zirconia before sintering is a useful method to increase surface roughness and could successfully improve the bonding strength of veneering porcelain.

ACKNOWLEDGMENTS

This study was supported by the National Natural Science Foundation of China (Grant No. 30872809 and No. 81371175).

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