Strength testing variables in dental ceramics
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CHAPTER 7

Staircase evaluation of the fatigue strength of sandblasted zirconia

Keywords: dental ceramics, zirconia, strength, fatigue, sandblasting
7.1 Abstract

**Objective:** The aim of this study was to assess the fatigue behavior of sandblasted zirconia using the staircase method in search of better performance.

**Methods:** 20 specimens for each group of a dental Y-TZP ceramic were polished or sandblasted with 50 μm or 120 μm alumina powders and subjected to cyclic or static fatigue loading. The specimen were loaded in a 3-point bending setup with 10,000 cycles at 1 s per cycle and a load time of 0.5 s, or with a constant load for 5000 s. The staircase method with 7.5% stress increments or decrements was used.

**Results:** Compared to the single load strength, the fatigue strength in cyclic tests was 86.3% for polished zirconia, 73.4% for sandblasted with 50 μm particles, and 42.3% with 120 μm particles. The fatigue strength in static tests was 85.9%, 78.5%, and 51.5%, respectively. Statistically significant differences were found between the surface treatments, but not between cyclic and static test.

**Conclusion:** Sandblasting with 50 μm particles produces significantly less degradation of the strength of this kind of zirconia than sandblasting with 120 μm particles.
7.2 Introduction
Dental restorations are exposed to the aggressive oral environment. Because of design and production related causes, these may fracture completely due to extreme incidental bite forces. Also the regular use causes fatigue-induced degradation and wear [1-3]. For all-ceramic restorations with a relatively brittle character it is a true challenge to withstand such odyssey. With the advantage of esthetics and biocompatibility many advanced ceramics have emerged in dentistry, which show improved strength [4, 5]. Currently, zirconia ceramics have been proved being the strongest and toughest commercially available all-ceramic core materials.

Zirconia has three crystallographic phases depending on the temperature and external pressure. The monoclinic phase is stable for pure zirconia at room temperature and pressure. The tetragonal phase exists at the temperature ranged between 1170°C and 2370°C while the structure becomes cubic at a temperature above 2370°C. So after pure zirconia is sintered at a temperature that is often at least 100°C higher than 1170°C, it undergoes a phase transformation during cooling from tetragonal to monoclinic phase starting at about 1170°C, which is accompanied by a volume increase of approximately 4.5%. However, dopant additions such as CaO, MgO, Y₂O₃, and CeO₂, etc. may stabilize the tetragonal phase at room temperature and can be involved in the favorable stress-induced transformation from tetragonal to monoclinic phase, which generate compressive stress, contributing to crack arrest and superior mechanical properties. With the introduction of zirconia in dentistry the era of the design and structural buildup of extensive all-ceramic bridges started. Nevertheless, zirconia ceramics are still brittle materials and have serious drawbacks; they have little tendency to deform, are rendered fragile by fairly small flaws, which are randomly distributed, and fracture quickly upon critical crack growth [1-5]. In fact, surface treatments such as milling, grinding, sandblasting, etc, which are routine procedures in the production of zirconia structures, may significantly compromise the strength [6-8]. Surface or near-surface damage from finishing procedures [9] would be great enough to initiate the crack from the flaws and accelerate the subcritical crack growth from the initial size to a critical size [10], which then jeopardizes of the structural integrity of the material, i.e. may cause fracture.

Sandblasting can introduce surface/near-surface defects and, hence, it could influence zirconia strength as present defects play a crucial role in initiating and driving crack growth under stress or stress intensity factor. It is hypothesized that sandblasting not only decreases the strength immediately but also compromises the fatigue behavior of zirconia. The purpose of the study was to assess the fatigue
strength of zirconia with different sandblasting surface treatment, using a staircase method. The zirconia tested in the present study was an yttrium-stabilized tetragonal zirconia polycrystals, Y-TZP, for dental use [5].

7.3 Materials and Methods

Zirconia (Cercon, Degudent GmbH, Hanau-Wolfgang, Germany) specimens were prepared in a same way in a previous study [7], from which also the single load values were used (Table 7.1). Milling zirconia blocks were cut in a sawing machine (ISOMET Q2 1000, Buehler Ltd, Lake Bluff, IL) using a diamond coated sawing disc into bars, which were sintered as recommended in a special oven (Cercon Heat, Degudent GmbH, Hanau-Wolfgang, Germany) for 6.5 hours at a maximum temperature of 1350°C. The sintered bars (17 mm x 2 mm x 1 mm) were polished with sandpapers in a sequence from 600 to 1200 grit (ECOMET Grinder/Polisher, Buehler Ltd, Evanston, Q3 IL). The surfaces that were to be exposed to tensile forces in the subsequent strength tests were sandblasted using either 50 μm or 120 μm alumina powder at 0.35 MPa pressure for 5 s at a distance of 20 mm (P-G 400/3, Harnisch+Rieth, Winterbach, Germany).

The specimens were mounted in a 3-point strength test holder immersed in distilled water. The 3-point setups were loaded cyclically in an air-driven fatigue machine (ACTA Cyclic Fatigue Tester, ACTA, the Netherlands). The valley-loading force was set as continuous at 5 N to keep the specimen from moving away from the original position. The peak-loading force was set according to ratio of the initial strength, i.e. single-load-to-failure strength, which was obtained from a previous study [7] (see Table 7.1). The cycle time was set at 1 s and the on-time at 0.5 s. The on-time load was kept at a same level during each loading. The specimens were subjected to $10^4$ load cycles. Based on a pilot study and the requirements for the staircase method, the peak loading force started at 80% of the initial strength for polished bars, 60% for sandblasted with 50 μm alumina, and 42.5% for sandblasted with 120 μm alumina. If a bar did not fail within $10^4$ cycles, the peak-loading force for the next one would be increased by 5%, or otherwise, decreased by 5%. 20 bars were involved in each group.

The static fatigue tests were carried out in a tensilometer (ACTA intense, ACTA, Amsterdam, NL) for three more groups with the same surface treatments and staircase procedure as in the cyclic fatigue tests. The static load was set up like the peak force for the cyclic load but kept without change for 5000 s. The calculation of the peak-loading force for cyclic test or the hold force for static test of an individual specimen was based on the normal 3-point flexure strength formula [11]:

\[
\text{Strength} = \frac{3PL}{2bh^2}
\]
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\[ \sigma_f = \frac{3WL}{2BD^2} \]

Where \( \sigma_f \) is the strength, \( W \) is the fracture load, \( L \) the support span, \( B \) the specimen width, and \( D \) the specimen thickness. The residual strength of the specimens, which had survived the cyclic and static staircase tests, was determined in the same 3-point setup.

The staircase result was analyzed with multiple logistic regressions [12]. The means and standard deviation of the fatigue strength were computed and compared with the ANOVA and Turkey’s pair wise multiple comparisons at a significant level of 0.05.

7.4 Results

In Figures 7.1 and 7.2 the staircase result of the cyclic and static fatigue are presented. The results are summarized in Tables 7.1 and 7.2. Statistic analysis of fatigue strength showed that there are no significant differences between the cyclic and static fatigue values with the same surface treatment, but all differences between the surface treatments are significant. Among these, the groups sandblasted with 120 \( \mu \)m alumina not only had the lowest fatigue strength (Table 7.1), but also presented the greatest decrease of strength compared to the single load strength and a much higher decrease than those sandblasted with 50 \( \mu \)m alumina or polished (Table 7.2).

Table 7.1 Fatigue, single load strength* (in MPa) and roughness* (Ra, Rp and Rv, in \( \mu \)m) with the standard deviations in parentheses.

<table>
<thead>
<tr>
<th>Loading</th>
<th>Surface</th>
<th>Polished</th>
<th>SB50</th>
<th>SB120</th>
</tr>
</thead>
<tbody>
<tr>
<td>Cyclic</td>
<td></td>
<td>1006.5 (60.1)</td>
<td>789.0 (103.9)</td>
<td>307.5 (6.0)</td>
</tr>
<tr>
<td>Static</td>
<td></td>
<td>1001.5 (121.9)</td>
<td>843.7 (121.4)</td>
<td>374.8 (80.4)</td>
</tr>
<tr>
<td>Single load*</td>
<td></td>
<td>1166.4 (192.3)</td>
<td>1074.6 (111.2)</td>
<td>727.5 (164.4)</td>
</tr>
<tr>
<td>Ra*</td>
<td></td>
<td>0.04 (0.00)</td>
<td>0.65 (0.06)</td>
<td>1.18 (0.11)</td>
</tr>
<tr>
<td>Rp*</td>
<td></td>
<td>0.17 (0.05)</td>
<td>2.77 (0.45)</td>
<td>5.22 (1.12)</td>
</tr>
<tr>
<td>Rv*</td>
<td></td>
<td>0.35 (0.16)</td>
<td>2.76 (0.29)</td>
<td>5.01 (0.71)</td>
</tr>
</tbody>
</table>

* data from reference [7]. SB50 / 120: sandblasted with 50 / 120\( \mu \)m alumina
Table 7.2  Ratio of the fatigue strength values to the single load strength

<table>
<thead>
<tr>
<th>Loading</th>
<th>Surface</th>
<th>Polished</th>
<th>SB50</th>
<th>SB120</th>
</tr>
</thead>
<tbody>
<tr>
<td>Cyclic</td>
<td>86.3%</td>
<td>73.4%</td>
<td>42.3%</td>
<td></td>
</tr>
<tr>
<td>Static</td>
<td>85.9%</td>
<td>78.5%</td>
<td>51.5%</td>
<td></td>
</tr>
</tbody>
</table>

SB50 / 120: sandblasted with 50 / 120μm alumina

Figure 7.1  Staircase results of the $10^4$ cycles fatigue tests (solid marks are surviving specimens).

Figure 7.2  Staircase results of the 5000 s static fatigue tests (solid marks are surviving specimens).

The mean residual strength values of the survived specimens are greater than the fatigue values of the same groups and similar for the cycling and the static tests. The residual strength of the polished and SB50 groups seems somewhat greater than
the initial (single-load) strength, whereas in the SB120 groups the residual strength is less (Table 7.3).

Table 7.3  Residual strength (in MPa) of survived zirconia specimens from staircase test with the standard deviations in parentheses

<table>
<thead>
<tr>
<th>Loading</th>
<th>Surface</th>
<th>Residual Strength</th>
<th>Ratio to single load</th>
<th>Ratio to fatigue</th>
</tr>
</thead>
<tbody>
<tr>
<td>Cyclic</td>
<td>Polished</td>
<td>1351.6 (120.9)</td>
<td>115.9%</td>
<td>134.3%</td>
</tr>
<tr>
<td></td>
<td>SB50</td>
<td>1253.4 (131.5)</td>
<td>116.7%</td>
<td>158.9%</td>
</tr>
<tr>
<td></td>
<td>SB120</td>
<td>604.8 (98.7)</td>
<td>83.2%</td>
<td>196.7%</td>
</tr>
<tr>
<td>Static</td>
<td>Polished</td>
<td>1227.6 (221.8)</td>
<td>105.3%</td>
<td>122.6%</td>
</tr>
<tr>
<td></td>
<td>SB50</td>
<td>1201.2 (139.2)</td>
<td>111.8%</td>
<td>142.4%</td>
</tr>
<tr>
<td></td>
<td>SB120</td>
<td>604.2 (105.5)</td>
<td>83.1%</td>
<td>161.2%</td>
</tr>
</tbody>
</table>

7.5 Discussion

An advantage of the staircase method is to give a good assessment of the mean fatigue strength and the feasibility of the computation of the standard deviation and statistical analysis [12]. In order to conduct a more severe simulating test, the specimens were submerged in an aqueous environment, which an important factor in mechanical aging of zirconia [28]. However, the loading cycles and the stress values in the present investigation were not representative for the long-term loading level of dental restorations in vivo. A relatively high cyclic peak stress for an accordingly small number of cycles was employed here, which is similar to maximum stress or low-cycle fatigue approach [13] and the outcome should not be used for a calculative prediction of the service life at a stress level in vivo. Yet, the current study demonstrates the sensitivity of zirconia to the particles size of sandblasting powders, which is consistent with the literature [14, 15]. The extrapolation can be made that at more dental stress levels, polished zirconia or sandblasted with 50 μm powder is much more resistant to fatigue degradation than zirconia sandblasted with 120 μm powder. Sandblasting with 120 μm powder not only reduces the (single load) strength, but decreases the fatigue strength to an even greater extent, which may yield very short service times of zirconia-based all-ceramic restorations in the oral cavity, presumably due to their severe surface/near-surface damage introduced by impact of large blasting particles [7, 14, 15].
According to Griffith’s theory and Weibull distribution [29] the residual strength of the survived specimens should perhaps be greater than the initial strength, as weaker specimens had been removed with the staircase tests. This is true for SB50 and polished zirconia, but the residual strength of the SB120 specimens is much less than the initial strength, which could be caused by stress corrosion in water.

Fatigue studies often find much greater differences between static and cycling tests, because of the breakdown of crack bridging / shielding ligaments behind the tip. These mechanisms progressively enhance the crack-tip driving force, and the wedging effect of fracture particles [21-23], which occur with cycling and are promoted at lower values of the stress intensity factor [24]. Such studies, however, use numbers of cycles, which are at least a hundred times greater and it could be that the small number of cycles in the current study and the consequently high loading force is largely responsible for the absence of difference between static and cyclic fatigue in the current study.

A purpose of this study was to illustrate the effect of surface treatments on fatigue life [16] rather than only subcritical crack growth (SCG) parameters and inert strength [17-20]. Predictions with computations based on data from SCG parameters and inert strength may be inaccurate where they fail to take the deleterious influence of certain surface treatments on (fatigue) strength into consideration. Inert strength has often been tested with artificial large surface defects and a high loading speed. Mechanical machining operations such as milling, grinding, polishing, sandblasting, etc. can result in different levels of surface damages and resistance to fatigue crack nucleation. Particle impact during sandblasting may induce potential surface/near-surface damage, like surface valleys acting as stress concentrator, nucleation of micro-defects, and the creation of microscopic, even dominant cracks, may be induced by [7, 9, 13-16], which is essential for the rate of the later advance of crack growth because the pre-existing defects are closer to the critical crack size [10]. Therefore, it becomes understandable that the condition of the surface has a decisive role in the initiation of fatigue cracks, which determine the service life, i.e. cause complete fracture. This susceptibility is revealed clearly in a very straightforward procedure with the staircase method [27] other than SCG methods.

Y-TZP does show some phase transformation, but it could be much more brittle compared to other stabilized zirconia such as Ce-TZP or Mg-PSZ. Although it shows the highest initial strength, it also has the lowest resistance against aging, shows a sudden drop of strength above a critical crack size and behaves rather like ordinary ceramics with a flat R-curve [25, 26]. Furthermore, the transformation in Y-TZP
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ceramics behaves in a different manner. The same zirconia, tested in a different study [30] showed much less transformation on the fracture surfaces than another Y-TZP, which suggests that the zirconia in the current study has less resistance to crack initiation and growth.

Being flaw-sensitive rather than tolerant, the yttrium stabilized zirconia tested in the present study is still a brittle material [25], which implies that during production procedures, attention and effort should be paid to control the condition of surfaces in an attempt to minimize the effect of fabrication-induced flaws. In previous studies sandblasting with 50 μm alumina improved the strength of milled and ground zirconia, possibly by removal of a damaged layer and creating a smoother surface, while sandblasting with 120 μm alumina was detrimental [7]. Also in the present study sandblasting with 50 μm alumina deteriorated the fatigue strength significantly less than 120 μm. Sandblasting with 50 μm alumina could be performed after sintering and before cementation. Diminishing flaws is helpful to delay the degradation of strength and prolong the functioning duration of zirconia-based restorations. Flaws should be eliminated and restorations treated gently to favor long-term clinical service.

7.6 Conclusion

Sandblasting with coarse particles, such as 120 μm alumina seriously degrades the fatigue strength of zirconia. Sandblasting with 50 μm particles is considerably less damaging and if sandblasting is necessary, is recommended.

7.7 References


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