Strength testing variables in dental ceramics

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CHAPTER 6

Strength influencing variables on CAD/CAM zirconia frameworks

*Keywords:* Zirconia; Particle abrasion; Surface damage; CAD/CAM
6.1 Abstract

Objective: Any studies in the dental literature look at the effect of different surface treatment methods on the flexure strength of zirconia where polished zirconia has been used as control. However, zirconia is subjected to different types of surface damage as a result of the CAD/CAM milling procedure and also to damage produced by other laboratory procedures in use daily. The aim of this work was to evaluate the effect of different surface treatment methods and in particular the effect of the CAD/CAM milling procedure on the flexure strength of zirconia frameworks.

Materials and methods: At least 20 zirconia bars (17 mm × 2 mm × 1 mm) for each group were prepared by either cutting and polishing zirconia milling blocks or by using a CAD/CAM device (Cercon) which left behind characteristic surface features related to the milling process. The fully sintered bars received either of the following surface treatments: air-borne particle abrasion (with 50 and 120 μm aluminum oxide particles, or both). Some bars received a heat treatment commonly used in baking veneer ceramics before or after particle abrasion. The surface roughness was measured for all bars, which were finally loaded in a three-point device. The fractured bars were examined using scanning electron microscopy. Data were analyzed using one-way analysis of variance and survivability was estimated using Weibull analysis (α < 0.05).

Results: There were significant differences in the flexure strength (in MPa) between the tested groups subjected to different surface treatments which can be categorized into four strength levels: (1074–1166 MPa) for polished zirconia and the CAD/CAM bars that were particle abraded (50 μm Al₂O₃) whether with or without heat treatment (936 MPa) for the ground bars that were particle abraded (50 μm Al₂O₃), (708–794 MPa) for CAD/CAM bars and for the polished bars that were particle abraded (120 μm Al₂O₃), and (546 MPa) for the ground bars that were particle abraded (120 μm Al₂O₃) being the weakest. There was a strong correlation between flexure strength and the severity of surface damage as indicated by surface roughness (R² = 0.912). Scanning electron microscopy revealed different types of surface and subsurface damage produced by the different surface treatments.

Conclusions: The surface damage produced by the CAD/CAM milling procedure significantly reduced the strength of zirconia which could be further weakened by different surface treatment methods resulting in unexpected failures at stresses much lower than the ideal strength of the material. It is advised to consider the effect of the CAD/CAM procedure on the characteristic strength when designing zirconia-based fixed partial dentures.
6.2 Introduction

The introduction of zirconia to the dental field opened up the design and application limits of all-ceramic restorations. The superior mechanical properties of zirconia combined with the state-of-the-art CAD/CAM fabrication procedure allowed for the production of large and complex restorations with high accuracy and success rate [1].

The strength of zirconia can be directly influenced by different surface treatment methods which exert different degrees and types of surface damage. These areas of surface flaws act as stress concentration sites and even though they are microscopic in nature, they act as potential sites for crack initiation and propagation [2]. Dental literature has focused on studying the effect of different surface treatments on the strength of zirconia-based materials and reduction in strength was generally associated with the degree of surface damage [3-5]. On the other hand, some studies reported an increase in strength after air-borne 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 [6-9].

A point worth noting is that in some of the previous studies polished zirconia was used as a reference point, while grinding with different grits of silicon carbide paper and air-borne particle abrasion at a high pressure were commonly used as examples of different levels of surface damage. On the other hand, under daily circumstances, zirconia is subjected to a different type of surface damage as a result of the milling procedure, which leaves behind characteristic trace lines and different patterns of surface damage and flaws [5]. Additionally, the combined effect of the CAM milling procedure and common laboratory procedures such as air-borne particle abrasion and multiple firing cycles used in baking the ceramic veneer, could be different from that expected for polished or disc-ground zirconia [10]. The mechanical properties and the long-term stability of CAD/CAM zirconia will be a function of the exerted surface damage, the degree of transformation, and the loading environment in terms of peak stresses and number of cycles [11].

The aim of this study was to evaluate the damage induced by the CAD/CAM milling procedure, combined with different surface finishing procedures, on the mechanical properties of zirconia and to analyze the interaction between these variables.
6.3 Materials and Methods

At least 20 zirconia bars (17 mm × 2 mm × 1 mm) for each group where prepared by either of the following methods: cutting zirconia milling blocks in a sawing machine (ISOMET Q2 1000, Buehler Ltd., Lake Bluff, IL) using a diamond coated disc saw (ground bars) or by using CAD/CAM technology (Cercon, Degudent GmbH, Hanau-Wolfgang, Germany) where individual bars were milled by the machine using wax replicas (CAM bars). The bars were sintered in the relevant manufacturer equipment (Cercon Heat uses a 6.5 h firing cycle at a maximum temperature of 1350 °C). Some of the ground bars were polished using ascending grit silicon carbide paper (ECOMET Grinder/Polisher, Buehler Ltd., Evanston, IL) up to 1200 grit. The ground or CAM bars received either of the following surface treatments:

1. Air-borne particle abrasion using either 50 or 120 μm aluminum oxide particles at 0.35 MPa pressure for 25 s/cm² at a distance of 2.0 cm. These bars were used to investigate the direct effect of air-borne particle abrasion on the mechanical properties of zirconia.

2. Some of the bars treated as above were subjected to one firing cycle used to bake the preformed ceramic veneer (1 min hold time in vacuum at a maximum temperature of 910 °C), either before or after air-borne particle abrasion (Austromat 3001, Dekema Dental-Keramiköfen GmbH & Co, Germany). These bars were used to investigate the effect of temperature on relieving surface pre-stresses caused by particle abrasion.

The surface roughness of the prepared bars was measured using a contact sensor (SJ-400, Mitutoyo Corporation, Japan) after which the bars were loaded in a three-point flexure device (15 mm span) at a cross-head speed of 0.5 mm/min in a universal testing machine (Instron 6022, Instron Limited, High Wycombe, UK). The load to failure was extracted from a computer-generated file and the flexure strength (MPa) was calculated using the relevant formula [12]. At least twenty bars were measured for each test group (n ≥ 20). Scanning electron microscopy (SEM) was conducted before and after flexure strength testing to examine the effect of the treatment applied to the surface structure and to analyze the fractured surface (XL20, Philips, Eindhoven, The Netherlands).

One-way analysis of variance and SNK post hoc tests were used to analyze the data. The failure probability under one-cycle loading was investigated using Weibull modulus. Pearson's correlation test was used to investigate the relation between flexure strength and surface roughness. According to the significance level (α = 0.5) and the
sample size \((n = 20)\), the test of choice had a power \((1 - \beta = 0.9)\) to detect large effect size differences \((F = 0.4)\), which in terms of flexure strength could be of clinical significance.

6.4 Results

Statistical analysis revealed significant differences in the three-point flexure strength values between the groups tested as a result of the different surface treatment methods applied \((F = 35.5, p < 0.000)\). According to strength values, the groups tested could be divided into the following categories: \((1074–1166 \text{ MPa})\) for polished zirconia and CAM and polished bars which were particle abraded \((50 \mu \text{m Al}_2\text{O}_3)\), \((936 \text{ MPa})\) for ground bars that were particle abraded \((50 \mu \text{m Al}_2\text{O}_3)\), \((708–794 \text{ MPa})\) for CAM bars, ground bars, and polished bars that were particle abraded \((120 \mu \text{m Al}_2\text{O}_3)\), and \((546 \text{ MPa})\) for the ground bars that were particle abraded \((120 \mu \text{m Al}_2\text{O}_3)\) being the weakest. Thermal firing either before or after particle abrasion, had no significant influence on the flexure strength (Table 6.1).

Pearson's correlation test indicated that there was a significant relation between the reduction in flexure strength and the severity of the surface damage induced by different surface treatments \((R^2 = 0.912)\). As indicated by the surface roughness measured, polished zirconia bars had the lowest surface roughness values, followed by particle abrasion with \(50 \mu \text{m particles}\), CAD/CAM defects, and particle abrasion with \(120 \mu \text{m}\) (Figure 6.1). The survival probability as calculated by Weibull modulus was also directly related to the degree of surface damage introduced by different surface treatments (Figure 6.2). The characteristic strength of the groups tested was calculated at 63% failure probability. A point worth noting is that CAD/CAM bars had the highest value of surface roughness, but on the other hand they were not the weakest compared with the other groups, which is explained in the following text.

Scanning electron microscopy of the fractured bars revealed that the critical crack always started from the surface flaws created and propagated to split the loaded bars in half. Compression curls were observed under the loading point and demarcated the shift from tensile to compressive fields. Air-borne particle abrasion resulted in severe surface damage as sharp scratches, cracks, grain pull-out, and material loss. The severity of this surface damage was more severe for the \(120 \mu \text{m}\) compared to \(50 \mu \text{m}\) particles. Additionally, the damage not only related to the surface, but extended to the subsurface region to a depth of few microns (Figure 6.3).
Table 6.1  Flexure strength (MPa) and surface roughness (μm) of the tested groups

<table>
<thead>
<tr>
<th>Surface condition</th>
<th>Roughness</th>
<th>Average strength a</th>
<th>S.D. of strength</th>
<th>Maximum strength</th>
<th>Minimum strength</th>
<th>Weibull modulus</th>
<th>Characteristic strength</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>$R_a$</td>
<td>$R_p$</td>
<td>$R_v$</td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>Polished</td>
<td>0.04</td>
<td>0.17</td>
<td>0.35</td>
<td>1166.39</td>
<td>192.28</td>
<td>1528.78</td>
<td>7.26 1244.17</td>
</tr>
<tr>
<td>Polished + SB120</td>
<td>1.18</td>
<td>5.22</td>
<td>5.01</td>
<td>727.54</td>
<td>164.44</td>
<td>1010.27</td>
<td>5.28 790.23</td>
</tr>
<tr>
<td>Polished + SB120 + fired</td>
<td>1.10</td>
<td>3.96</td>
<td>5.00</td>
<td>760.72</td>
<td>122.51</td>
<td>1026.16</td>
<td>7.50 809.92</td>
</tr>
<tr>
<td>Polished + SB120 + fired + SB120</td>
<td>1.11</td>
<td>4.29</td>
<td>4.66</td>
<td>45.99</td>
<td>160.36</td>
<td>1041.98</td>
<td>5.38 809.22</td>
</tr>
<tr>
<td>Polished + SB50</td>
<td>0.65</td>
<td>2.77</td>
<td>2.76</td>
<td>1074.57</td>
<td>111.21</td>
<td>1228.06</td>
<td>9.91 1130.70</td>
</tr>
<tr>
<td>Polished + SB50 + fired</td>
<td>0.62</td>
<td>2.73</td>
<td>2.60</td>
<td>1080.67</td>
<td>103.95</td>
<td>1213.40</td>
<td>11.80 1128.15</td>
</tr>
<tr>
<td>Polished + SB50 + fired + SB50</td>
<td>0.59</td>
<td>2.86</td>
<td>2.60</td>
<td>1076.09</td>
<td>118.73</td>
<td>1233.23</td>
<td>10.58 1127.94</td>
</tr>
<tr>
<td>Ground</td>
<td>1.18</td>
<td>6.51</td>
<td>4.38</td>
<td>94.83</td>
<td>156.47</td>
<td>1173.47</td>
<td>6.13 855.79</td>
</tr>
<tr>
<td>Ground + SB120</td>
<td>1.22</td>
<td>4.18</td>
<td>6.27</td>
<td>546.56</td>
<td>103.58</td>
<td>753.00</td>
<td>6.19 588.48</td>
</tr>
<tr>
<td>Ground + SB50</td>
<td>0.66</td>
<td>2.63</td>
<td>2.87</td>
<td>936.13</td>
<td>211.56</td>
<td>1321.06</td>
<td>5.08 1018.56</td>
</tr>
<tr>
<td>Polished + SB120 + SB50</td>
<td>0.87</td>
<td>3.10</td>
<td>3.50</td>
<td>708.72</td>
<td>125.55</td>
<td>951.51</td>
<td>6.74 758.93</td>
</tr>
<tr>
<td>CAM</td>
<td>1.91</td>
<td>8.99</td>
<td>7.20</td>
<td>743.35</td>
<td>213.74</td>
<td>1084.46</td>
<td>3.99 820.65</td>
</tr>
<tr>
<td>CAM + SB50</td>
<td>1.61</td>
<td>5.81</td>
<td>6.33</td>
<td>1123.93</td>
<td>135.10</td>
<td>1399.09</td>
<td>9.99 1180.92</td>
</tr>
</tbody>
</table>

SB120: sandblasted with powder of a mean size of 120 μm; SB50: sandblasted with powder of a mean size of 50 μm; CAM: as machined by CAM/DAM device. a Average flexure strength of the groups with same superscript letter was not statistically significantly different ($F = 35.5, p < 0.001$).
**Figure 6.1** Correlation between flexure strength and surface roughness.

**Figure 6.2** One-cycle load failure probability of zirconia bars with different surface treatments.
Figure 6.3  (A top) Grain pull-out, sharp scratches, and pitting after particle abrasion with 120 μm aluminum oxide. (B bottom) Deep sharp crack (30 μm) as a result of particle abrasion with 120 μm aluminum oxide on ground zirconia.
Figure 6.3  (C top) Critical crack originated from the damaged surface as result of particle abrasion with 120 μm aluminum oxide. (D bottom) Cross-section view demonstrating the subsurface damage induced by particle abrasion with 50 μm aluminum oxide.
Figure 6.4  (A top) Trace lines left on the surface as a result of CAM milling procedure. The observed macroscopic features were not effective as crack initiation sites. (B bottom) Oblique image demonstrating that the critical crack (white arrow) was not related to CAM milling trace lines (black arrow).
Figure 6.5 Severe damage introduced by grinding zirconia.

On the other hand, the CAM milling procedure resulted in the creation of milling trace lines, which in the present research were parallel to the long axis of the bars. These trace lines did not act as crack initiation sites (Figure 6.4). Additionally, other milling defects, such as premature contact with the milling burs and detached islands of zirconia grains, were also observed all of which resulted in the large variations in the measured surface roughness and naturally resulted in a large standard deviation for the strength measured for this group. Grinding zirconia frameworks before sintering (ground bars) resulted in severe damage to the surface in the form of large islands of detached zirconia grains and deep defects (Figure 6.5).

6.5 Discussion

As the main function of the underlying framework is to support the ceramic veneer and to carry the loading forces, different laboratory tests were used to evaluate the internal strength of zirconia frameworks. The design of fixed partial dentures can be considered as a simple beam and different flexure strength tests are frequently used for strength evaluation [4]. On the other hand, standard flexure strength tests do not take into account important factors such as the effect of design, variation in thickness of the framework, and the nature of human occlusion and loading environment [13]. Nevertheless they offer a controlled environment for evaluation of the interacting variables of interest.

The strength of all-ceramic materials is directly related to the size, population, and distribution of structural defects and flaws. Additionally, the location of these
defects also plays an important role. While bulk defects are more shielded and protected by the surrounding material, surface defects directly act as stress concentration sites, which magnify the applied stresses according to the severity of the surface flaw [14]. This severity is a function of the size and shape of the surface defect. Sharper and deeper defects increase the concentration of stress at their crack tips and thus are more likely to act as crack initiation sites [15].

Achieving a flaw-free state of a material seems only possible in theoretical applications as each material has some characteristic flaw population related to the fabrication and handling procedures selected. On the other hand, fine polishing tends to reduce the severity and the population of surface defects and flaws to a degree where the internal strength of the material becomes the dominant factor determining its mechanical performance [4]. According to the results of this study, the flexure strength values associated with the different surface treatment methods used could be categorized into four groups with polished zirconia being the strongest (Table 6.1).

It is clearly depicted in Figure 6.1 that there is a direct relationship between variations in the flexure strength and the associated surface roughness for each test group (Pearson correlation factor = 0.912). Three parameters were selected to analyze surface roughness, the $R_a$ value which describes the average surface roughness as a mean of the elevations and depressions measured from an estimated surface, the $R_p$ value which represents the average vertical elevations measured from the estimated average surface, and the $R_V$ value which is the surface depressions measured from the estimated surface and in this case was the most representative of the resultant surface flaws and defects acting as effective stress concentration sites. The presented data are in agreement with Luthardt et al. who also reported similar surface roughness values but relatively lower flexure strength for ground zirconia [4].

The only apparent misleading deviation from this linear relation is that CAM bars were associated with the highest average surface roughness ($R_p = 8.9 \mu m$) and at the same time were not the weakest. This could be explained by the fact that the milling procedure was executed before the final sintering, while the other surface procedures tested were carried out after the final sintering. The sintering procedure might have a healing effect on the surface damage caused by the CAM grinding procedure. Moreover, the CAM milling burs have a circular cross-section and they contact the surface of the framework in calculated plans and contact points leaving behind milling trace lines (Figure 6.4). These macroscopic surface roughness or elevations do not directly act as stress concentration sites, but in fact it is the
microscopic surface roughness represented by sharp cracks and scratches that act most effectively as crack initiation sites [16].

While even the smallest damage introduced either by the CAM milling procedure or particle abrasion may at the beginning seem trivial, it will have a detrimental effect on the fatigue life of zirconia restorations. With repetitive cyclic loading during mastication, small defects tend to grow in size until reaching a critical size where catastrophic failure will eventually result. Thus any temporary beneficial effects induced by different surface treatment methods will be counter weighted by crack growth phenomena [17, 18]. With consideration that crack growth starts at a threshold intensity factor, which for zirconia ($K_{I0} = 3.1 \text{ MPa m}^{1/2}$) [19] is much lower compared to its critical crack intensity factor ($K_{ic} = 7.4 \text{ MPa m}^{1/2}$) [2], the smallest surface defect could be large enough to act as an effective stress concentration site, finally increasing the fracture probability [20].

In partial agreement with other studies which reported an increase in flexure strength after particle abrasion, relating such findings to the creation of a compressive field as a result of tetragonal–monoclinic transformation [6-9], the data of this study indicate that particle abrasion with 50 $\mu$m aluminum oxide resulted in an increase in the strength of CAM and ground bars, possibly by removing weakly attached surface grains and by the elimination of milling and grinding trace lines [6]. Supporting such explanation is the reduction of the surface roughness observed after particle abrasion (Table 6.1). On the other hand, if the increase in flexure strength was due to phase transformation, such effect would be lost after thermal firing which was not the case in this study. On the contrary, particle abrasion with 120 $\mu$m aluminum oxide resulted in significantly weakening all the bars tested and in increasing the surface roughness, which also explains such a deteriorating effect. Even though the strength of the CAM or ground bars that were air-borne particle abraded with 50 $\mu$m aluminum oxide was higher compared with the non-abraded bars, the long-term effect of the induced damage should be considered.

In addition to surface roughness, thermal firing of the framework before or after air-borne particle abrasion was investigated. While other studies found that thermal firing after particle abrasion lowers the strength of zirconia and related such finding to the reverse of the monoclinic phase back to tetragonal, which relieves the created compressive fields [6], in the present work thermal firing had no influence on the flexure strength whether performed before or after particle abrasion but on the other hand it resulted in an increase in the Weibull modulus of the air-borne particle abraded polished zirconia either with 50 or 120 $\mu$m particles from 9.91 to 11.8 and from 5.28 to
7.5, respectively. As zirconia is a glass-free material, whether this is related to reverse transformation or due to relieving any present pre-stresses, it remains a point for further investigation [6, 21, 22].

The probability of the fracture of zirconia under a single-load cycle (Figure 6.2) indicates that zirconia is a highly reliable material for construction of all-ceramic frameworks and it can handle surface damage parameters, which if introduced to other ceramics would result in catastrophic failure at much lower loads. Nevertheless, one should not overestimate the strength of zirconia, as catastrophic failure could result at extremely unexpected low loads, 379 MPa minimum strength for ground zirconia with 120 μm particle abrasion.

Bearing in mind that CAD/CAM fabricated zirconia frameworks are already weakened by the surface damage induced by the milling procedure, all strength calculations should be redesigned using the characteristic strength of CAD/CAM fabricated zirconia, 820 MPa instead of 1244 MPa as calculated for polished zirconia. Careful selection of the surface treatment method of choice, which does not induce further excessive surface damage, is a prerequisite for the success of zirconia restorations.

6.6 References


