IMPACT OF SURFACE ROUGHNESS ON FLEXURAL STRENGTH AND FRACTURE TOUGHNESS OF IN-CERAM ZIRCONIA

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ABSTRACT

Objective. In clinical practice, core material can be exposed after adjustments, which may result in loss of ceramic glaze. The objective of this study is to evaluate the effect of surface roughness on the flexural strength and fracture toughness of In-Ceram zirconia core material. Materials and methods. Forty In-Ceram zirconia specimens were prepared and divided into two main groups (n=20) according to the type of mechanical test. Group 1, for flexural strength test and Group 2, for fracture toughness test. Each group was further subdivided into two subgroups (n=10) according to type of surface treatment. Subgroup A, glazed while subgroup B, finished and polished. Qualitative evaluation of two representative specimens of each subgroup has been done with the assessment of SEM. The flexural strength and the fracture toughness tests were determined. Statistical significant among groups was analyzed using Mann-Whitney test and Kendall’s tau test. Results. Glazed subgroups had the smoothest surface, while the finished and polishing subgroups had the coarsest. Glazed subgroups demonstrated significantly the higher mean flexural strength values (385.38±45.42) and the higher fracture toughness values (6.07±1.00). Conclusion. Surface roughness has adversely affected the flexural strength and the fracture toughness of In-Ceram zirconia.

INTRODUCTION

All-ceramic dental materials are becoming the first choice of restorative materials because of their superior biocompatibility and distinct aesthetic appeal. However, brittle behavior combined with extreme sensitivity to micro crack-like defects has hampered their wider use and limited their application [1-3]. Flows and defects that may grow at the microscopic level have been shown to significantly control their strength characteristics [4].

The In-Ceram has been developed using an aluminum oxide slip casting technique to build the framework which is then fired to an open-pore microstructure. The material gains its strength by infiltration of the open-pore In-Ceram alumina microstructure with lanthanum glass [5]. The high flexural strength of glass infiltrated In-Ceram alumina material (400-605 MPa) depends on the strength of the fired bond between the aluminum oxide particles and the complete wetting of the open-pore...
microstructure by lanthanum glass infiltration [6].

The mechanical properties of In-Ceram alumina can be improved by adding Zirconium oxide [7]. The addition of 33 wt% of zirconium oxide leads to an increase of the flexural strength up to 750 MPa, while fracture toughness is doubled [8].

The cores must be veneered with suitable ceramics and are not intended to be exposed in dental use [9]. However, adjustment to improve occlusion by grinding of luted all-ceramic restorations can expose the core. The ceramic restorations have to be adequately glazed before placed in the oral cavity. Non-glazed ceramics have been shown to cause undesirable responses to adjacent periodontal tissues, increase the potential of wearing opposing teeth, and to be more susceptible to plaque accumulation [10-12]. Moreover, surface roughness can influence its mechanical properties [13, 14].

The processing procedures and/or clinical adjustments are more likely to initiate sub critical flaws or large defects which, up on clinical loading and/or presence of moisture, may grow to a critical situation leading to catastrophic failure [15]. In addition, different surface roughness formed through different finishing procedures can cause various stress concentrations and consequently may be accompanied by a reduction in strength [16]. The superficial roughness must be minimized with use of polishing techniques to achieve an acceptable smoothness, this means to make the material as inert as possible [17]. The effect of the processing procedures, polishing, finishing and glazing on the mechanical properties of some dental materials has been studied by many investigators [18-24]. However, there is controversy concerning the most suitable method that can produce a smooth and strong surface.

The aim of this study was to evaluate the effect of different surface treatment procedures, glazing and finishing + polishing, on the flexural strength and fracture toughness of In-Ceram zirconia core material and to test the hypothesis that its strength is dependent on surface roughness.

### MATERIALS AND METHODS

Forty In-Ceram zirconia specimens (VITA Zahnfabrik, Bad Saeckingen, Germany, Shade CE 0124) were fabricated for this study according to the manufacturer’s specifications.

#### Impression Making.

A copper mold was specially designed with a central depression of (20, 3, 4 mm) in dimensions. An impression of the mold using an addition silicon impression material (Swiss Tec, Coltene Whaledent, 9450 Altstatten / Switzerland). The copper mold was placed over the impression material, held under finger pressures till complete setting, then removed and poured with In-Ceram special plaster (Vita In-Ceram, Vita Zhanfabrik spitalgasse 3 Germany). After complete setting of the plaster it was painted with a special isolating gel (Vita isolating gel).

#### Ceramic Fabrication.

In-Ceram zirconia powder was mixed with the mixing fluid into a glass beaker according to manufacturer’s instructions. The powder slip was added and spatulated into the liquid in a small portions while the beaker was placed on a vibrator, the beaker was placed in an ultrasonic unite for 7 minutes to ensure homogeneous non-sticky mix then placed in a vacuum for one minute.

The slip was built up in the working plaster model, then placed in a furnace (Vita Vacummat 200) for ½ hour in a temperature ranging from 130-190 °c until shrinkage occurs to the plaster then, the ceramic specimens separated and placed in the furnace for 40 minutes as holding time at 1120’c.

The specimens were glass infiltrated and placed in the furnace for 40 minutes holding time and then subjected to another cycle for 5 minutes at 1100’c for glass controlling.

Finally the specimens were cleaned using an ultrasonic bath with acetone at room temperature for 15 minutes.

The final sizes of the specimens for flexural strength test was (20mm length, 1.5 mm thickness, and 4mm width) while for fracture toughness was (20mm length, 3mm thickness, and 4mm width).
### Grouping of Specimens:

Specimens were then divided into two groups \( n=20 \) according to their size.

**Group 1**, for flexural strength test and **Group 2**, for fracture toughness test. Each group was further subdivided into two subgroups \( n=10 \) according to type of surface treatment.

**Subgroup A**, glazed by heating the specimens to the glazing temperature prescribed by the manufacturer and holding it at that temperature for 5 minutes.

**Subgroup B**, was submitted to wet finishing with diamond bur \( (# 4138 \, KG, \, Sorensen) \) followed by wet polishing with silicon rubber polisher in different grains\(^{[16]}\).

The thickness of each specimen was measured at three different positions and the mean value was recorded. All the specimens were then cleaned using ultrasonic bath for 15 minutes and washed in boiling water.

### Scanning Electron Microscopic Examination:

Two representative specimens from each subgroup of In-Ceram zirconia were sputter coated with 300-500 Å gold and examined using SEM (JEOL 5300, JMST-20,19 K.V., Japan). This examination was utilized to qualitatively evaluate the effect of glazing and finishing +polishing on the surface of the specimens with the help of photomicrographs.

### Uniaxial flexural strength:

A universal-testing machine (Type 500, Lloyd instrument, city, England), was used for the flexural test. The rate of load application was adjusted to 2mm/min. The test specimens was placed centrally between a self aligning fixture the edges of the specimen were about 2mm from the fixture, leaving the test span (center-to-center between bearers) to be 16mm. Load was applied perpendicular to the long axis of the test piece (Fig1). The flexural strength \( M \) for the specimen was calculated in Newtons per square meter (MPa) according to equation (1)

\[
M = \frac{3WL}{2bd^2}
\]

![Image](image.png)

Fig(1): Flexural strength test speacimen in the universal testing machine.

Where \( W \) is the breaking load (N); \( l \) is the test span (mm), \( b \) is the width of the specimen(mm); \( d \) is the thickness of the specimen.

### Fracture toughness:

A notch of the specimens was cut with a 0.1 mm thickness diamond saw disc. The saw depth was nearly half of the specimen width \( W \). fracture toughness was measured with single-edge notched beam method SENB\(^{[25]}\). Immediately before testing the width, thickness, and span length of the specimen were measured. Fracture toughness test was done as a special fixture for three point bending rest with the notch of specimen facing down and the distance between the two support was kept constant at 16 mm throughout the testing. A universal testing machine (Type 500, Lloyd instrument, city, England) was used for the application of a compressive load at a cross-head speed of 2mm /min. until fracture of the specimen occurred. The load value at fracture was recorded and the critical stress intensification factor, fracture toughness \( K_{IC} \) value was calculated in MPa.m\(^{1/2}\)) for each specimen according to equation (2):

\[
K_{IC} = \frac{PS}{Bw^{3/2} \times (a/w)}
\]
Where $P$ is the load value; $S$ is the test span (mm), $B$ is the thickness of the specimens (mm), $W$ is the width of the specimens (mm), and $a$ is the crack length.

**Statistical analysis:**

The data were compared for all groups using Mann-Whitney test. A $P$-value of $<0.05$ was considered statistically significant. And the correlation between flexural strength and fracture toughness in all tested specimens was done using Kendall’s tau test.

**RESULTS**

The SEM examination of the specimens shows a characteristic rough spots due to finishing + polishing and exhibited a more heterogeneous surface condition (Fig 2) compared with the glazed specimens (Fig 3) which presented the smoothest topographic pattern.

Means, standard deviations of flexural strength and fracture toughness of the two main tested groups are listed in Table 1.

The glazed In-Ceram zirconia specimens showed the highest value of the flexural strength ($385.38 \pm 45.42$), and also the highest value of the fracture toughness ($6.07 \pm 1.00$)

The finishing + polishing of In-Ceram zirconia specimens showed the lowest value for both flexural strength and fracture toughness ($302.38 \pm 47.55$) and ($2.14 \pm 0.46$) respectively.

Mann-Whitney test is used for statistical analysis, the glazed In-Ceram zirconia specimens showed a higher statistically significant difference $P=0.002$ among the mean flexural strength values of the two tested subgroups. And also the glazed In-Ceram zirconia specimens showed the highest statistical significant difference $P<0.001$ among the mean fracture toughness of the two tested subgroups.

It was found that there is a significant positive correlation between flexural strength and fracture toughness in all tested specimens ($Kendall’s\tau b = 0.467 \& P=0.012$).

**TABLE (1)** Statistical analysis of surface roughness of In-Ceram zirconia and its effect on flexural strength and fracture toughness using Mann-Whitney test:

<table>
<thead>
<tr>
<th></th>
<th>Glazing</th>
<th>Finishing + polishing</th>
<th>$P$</th>
</tr>
</thead>
<tbody>
<tr>
<td><strong>Group I</strong></td>
<td>$385.38^a$</td>
<td>$302.38^b$</td>
<td>0.002</td>
</tr>
<tr>
<td></td>
<td>$\pm 45.42$</td>
<td>$\pm 47.55$</td>
<td></td>
</tr>
<tr>
<td><strong>Group II</strong></td>
<td>$6.07^a$</td>
<td>$2.14^b$</td>
<td>0.001</td>
</tr>
<tr>
<td></td>
<td>$\pm 1.00$</td>
<td>$\pm 0.46$</td>
<td></td>
</tr>
</tbody>
</table>

Different superscript letters ($a, b$) indicate groups that are significant different ($p \leq 0.05$)
DISCUSSION

The analysis of the effect of surface roughness of a ceramic core material on failure stress is important, since almost every dental restoration is selectively ground chair-side to avoid occlusal interferences. The goal of finishing and polishing procedures is to obtain the desired anatomy, proper occlusion and the reduction of roughness that were produced by the contouring and finishing instruments. The polished surface should be smooth enough to be well tolerated by oral soft tissue and to resist bacterial adhesion and excessive plaque accumulation [26, 27].

The failure of many materials, including ceramics, has been attributed to the propagation of a large system of densely distributed cracks, rather than to a single precisely defined fracture [28, 29].

Fracture toughness is extremely sensitive to the applied methodology. Amongst a variety of techniques such as chevron-notch-beam test (CNB), indentation strength (IS), the indentation fracture technique (IF), single edge notched beam (SENB) has been selected which is the only statistically comparable method to (CNB) which is the more accurate technique [25].

The analysis of Figs 2 and 3 showed that their topographic patterns are different these results were assessed qualitatively using SEM. Based on these data, it may be stated that finishing and polishing was not able to reestablish an adequate surface roughness in comparison to the original glazed surfaces.

The results of the current investigation exhibited marked increase in the values of both flexural strength and fracture toughness in Glazed In-Ceram zirconia specimens than that of finishing and polishing. The rougher the surface, the lower were the flexural strength and the fracture toughness of the specimens. This could be attributed to the fact that glazing is believed to increase the strength of the ceramic materials by reducing the depth and / or sharpness of critical flaws. These results are in agreement with that obtained from the study of Lohbauer et al [30] who suggested that the ceramic core material exhibited a substantial decrease in flexural strength and fracture toughness with increasing surface roughness. Our finding is also in good agreement with those of Jager et al [16] in which this correlation was also found on dental ceramics, and they concluded that, glazing could round the crack tip of possible micro cracks, thus it will avoid creation of the surface roughness that will consequently result in non uniform stress distribution and local concentration of the stress.

This relation between the flexural strength and surface roughness supports the hypothesis that surface roughness will concentrate an applied stress resulting in lower flexural strength.

In contrast with the results of the current study Giordano et al [33] &Chu et al [34] concluded that, the hypothesis that the strength is affected by surface roughness is not accepted. They explained also that, the stress concentration can be initiated not only from the surface roughness but also from other factors such as internal stress, porosity, inherently developed cracks and thin sectional areas close to tensile stresses.

The results of this study found that there is a positive correlation between flexural strength and fracture toughness in all tested specimens this was in agreements with the results of other investigations [31, 32].

CONCLUSIONS

Within the limitations of this in vitro study the following conclusions were drawn:

1. Finishing and polishing of In-Ceram zirconia core material results in more rougher surfaces than that produced with the glazing procedure.
2. In-Ceram zirconia core material exhibited a significantly lower flexural strength and fracture toughness with finishing and polishing.
3. There is a significant positive correlation between flexural strength and fracture toughness.
REFERENCES


